

A LABORATORY OUTLINE  
OF  
GENERAL CHEMISTRY

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ALEXANDER SMITH

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# A LABORATORY OUTLINE OF GENERAL CHEMISTRY



BY


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SECOND EDITION    REVISED

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## PREFACE TO THE FIRST EDITION.

No apology is offered for the preparation of another laboratory manual. Each teacher of chemistry sooner or later catches the infection, and finds himself impelled to prepare an outline of his own. There is little material that is original in the present one. The author, therefore, acknowledges his indebtedness to other similar outlines. Ramsay's "Experimental Proofs of Chemical Theory," may be named as the source of some of the quantitative experiments.

In selecting, applying, and, to a slight extent, adding to this material, several considerations have been kept in view, although the nature of the case has made it easier to give effect to some of these than to others.

The laboratory work is most emphatically not a mechanical part of the course, in which the use of the intelligence of the student plays no part and all thought is reserved for the home or class-room. It is an essential part of the rigorous study of the subject, requiring the employment of the head as well as the hands. An effort has therefore been made to give continuity to the directions by the form in which they are given, and, by questions, to prepare the way for the correlation of the facts which is accomplished in quiz and lecture. This does not mean that the work forms by itself a complete course of study in the subject. On the contrary, certain important topics, such as Gay-Lussac's "Law of Volumes" with the inferences, based on Avogadro's hypothesis, which may be drawn from it, can more fitly be illustrated in the lectures. The admirable experiments of Hofmann often involve numerous details and precautions in manipulation the need of which the beginner could not have foreseen. The apparatus is difficult to handle on the first trial and its provision involves the teacher of a large class in difficulties. The significant facts to be observed in each case are so very simple as not absolutely to require individual experiment for



their comprehension. These facts, however, demand elaborate reasoning for the development of their significance, and anything like spontaneous application by the pupil is very unlikely to occur. Thus the difficulties preceding and following the observation of the central fact render it unsuitable for illustration in the laboratory. On the other hand, when the practical details are subordinated, the diagrammatic nature of these experiments renders them peculiarly adapted for lecture purposes, for which, in fact, they were originally designed. Then, too, the measurement of volume appeals to the eye more directly than the measurement of weight and is, therefore, more suitable for the lecture room. The measurement of weight, on the other hand, is more convincing when carried out by the pupil himself and is, therefore, better adapted to form the basis of the majority of the quantitative laboratory experiments.

Many other cases of the omission of familiar experiments, with a view to their utilization in the lecture, will be noted.

The science of chemistry deals with natural phenomena themselves, and not what various authors have said about them. It is the part which the pupil can learn first-hand in the laboratory by observation and inference, and not the part, indispensable though it is, which he borrows from the book or teacher which will correctly shape his attitude toward the whole science. Much opportunity for induction has therefore been provided. This aspect of the work has been emphasized by an effort to discredit reliance on books. For this purpose experiments with easily ascertainable results, whose course nevertheless cannot be anticipated by reference to the usual run of text-books, have been introduced occasionally. Such are the action of concentrated sulphuric acid on ferrous sulphide (p. 57), and of magnesium on nitric acid (p. 68).

There is an absurdity inherent in the usual course of directing a pupil to make a single qualitative experiment, with haphazard proportions of the materials and other conditions largely selected by chance, and then asking him to write an equation, that is, to draw a quantitative conclusion.

The pupil feels this distinctly, although he may attribute it to the wrong cause. . . . It is not always convenient to follow quantitatively every feature, even in one of these selected chemical changes, but some features may be investigated closely in one, and others in another, with a resulting synthesis of the whole process in the mind of the pupil. Thus in the burning of phosphorus the composition of the pentoxide cannot be measured by the beginner. Again, in the determination of the combining weight of iron with oxygen (p. 16), the action of the nitric acid on the iron and the effect of heating the nitrate are passed over to avoid premature discussion of the properties of nitric acid. The actions of carbon on oxygen (pp. 12 and 15), and of zinc on hydrochloric acid (pp. 17 and 18), are examples of the more complete investigation of a chemical change.

Even in cases where the quantitative aspect is deliberately left to be supplied by the book, there are often essential factors in the material from which a conclusion is to be drawn which are, at the time, unknown to the student and beyond his observation. Where these occur a direct reference to the text-book is enjoined by inserting an R in parentheses in the directions. Thus in Chap. IV (oxygen), 3 and 4, the interaction of certain anhydrides with water, the solubilities of nitrates, chlorides and chlorates of potassium and silver, etc., are involved in the understanding of the chemical changes observed. This sign is intended to enable the pupil to distinguish when he is expected to rely on himself and when he must seek aid, while being held responsible for reaching the correct conclusion in either case. To carry out this idea, as well as on other grounds, a few volumes for reference (including a table of solubilities) should be provided in the laboratory.

Experiments have been inserted to illustrate such theoretical matters as the measurement of the strength of acids and phenomena of chemical equilibrium, ionic equilibrium, solution tension, etc., since these ideas cannot be grasped in such a way as ever to be applied unless they are interwoven with the facts they explain. Their introduction requires no justi-



fiction. Physical chemistry cannot be taught in the first year of general chemistry, but its results are absolutely indispensable to a rational correlation and explanation of the facts dealt with in general chemistry.

In Chaps. I–IX, which form one-third of the year's work, the lectures precede the laboratory work, while not covering altogether the same ground. In this way the beginner acquires some knowledge of the nature of the substances he is to handle, and of the sort of results he is to look for. In Chaps. X–XXV, the lectures follow the laboratory work and the latter is therefore more strictly inductive than in the earlier chapters.

The work outlined is designed to occupy about six hours of laboratory work per week for nine months, on the assumption that a proportion of the experiments is selected for omission or transference to the lecture room. The order in which the elements are presented and the period at which quantitative work is introduced may readily be changed as circumstances demand.

College students who have already completed a year of chemistry in a secondary school will naturally have performed many of the experiments here given, and have learned all they can teach. A selection, by way of suggesting a basis for a college course in continuation of school work for such students, is printed in the Appendix. . . .

I have to thank Professor J. B. Garner (Wabash College), and Dr. H. N. McCoy (of this department) for many valuable suggestions. I owe to the latter also the admirable working out of the details of the quantitative experiments on the composition of carbon dioxide (p. 15), on the proportions of copper to oxygen in the two oxides (p. 19), on the combining weight of iron (p. 16), and on the estimation of the activity of an acid (p. 64), as well as the realistic arrangement for making sulphuric acid from pyrite (p. 59). Finally I have to express my indebtedness to Miss Carol Paddock for preparing the illustrations.

Chicago, May, 1899.

ALEXANDER SMITH.



## PREFACE TO THE SECOND EDITION.

In preparing this edition the plan of the former one has been preserved and its application has been made more thoroughgoing. An almost entirely new set of figures, some new experiments, and innumerable alterations in the wording of the directions and the questions have been introduced. The chief objects in view have been to secure a more logical presentation of the subject and to make the student less dependent on help from the instructors.

The experiments have also been adapted more closely to the set of apparatus in the possession of each student. Except in cases of breakage, it should not now be necessary for him to visit the storeroom more than a dozen times in the course of the whole year.

The teacher will find it a great help to furnish his students with a carefully compiled list of references, using books in his particular laboratory. Without this much time may be wasted through ignorance of where to look for the material required in answering questions to which an [R] is appended.

As some of the opinions expressed in the preface to the first edition have since appeared in expanded form in *The Teaching of Chemistry and Physics*, written by the author in collaboration with Professor E. H. Hall, this preface has not been reprinted in full. The presence of most of it, however, seemed to be called for in explanation of certain features of the present book.

Chicago, October, 1902.

A. S.



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## GENERAL INSTRUCTIONS.

**Check your apparatus** by comparison with list, have deficiencies made good at the storeroom, obtain the key of a locker in the hall, enter the numbers of this and the desk keys on the list, and then sign and return the list to the storekeeper.

Read the "Regulations" posted in the laboratory.

Provide yourself with a **notebook** and make a careful permanent record immediately after each experiment. Enter the numbers of the chapters and paragraphs of the outline systematically, so as to save the necessity of copying the directions, and place the same numbers at the head of each page in the notebook. State (1) what you did, if anything beyond the directions, (2) what you observed, (3) what conclusions you drew. A sketch of the apparatus will enable you to recall the circumstances of the experiment, if later reference to it is necessary.

The **blank pages** are not intended for the final notes. They may be used for individual suggestions given by the instructor, preliminary notes, record of weighings, etc.

Whenever an **interrogation point** (?) or a **direct question** appears, a corresponding note should appear in the notebook.

The very numerous **questions** asked in the course of this outline are intended to be answered, not by speculation, but by careful observation, and reasoning based on the results of this. In many cases the student will find it necessary to devise and carry out further experiments of his own before a satisfactory answer is obtained. In some more complex cases, where much time and work would be necessary, the books set apart for reference are to be consulted. An [R] indicates such necessity. This should be done, however, only after the experiments have been made and the notes written up as far as possible.

The directions have been expressed with the utmost care and brevity. Every word is significant. *Italics* are therefore nowhere employed.

The equation should always be given in the notes when a chemical change has been observed, but an equation alone is never a sufficient record.

Where the word [Instructions] appears, consult the instructor before going further.

In experiments marked [Quant.] use the finer balance, in all other cases the rough scales in the laboratory.

The expression [Temp. order] indicates that the necessary apparatus must be obtained from the storeroom on a temporary order.

Where exact quantities are not indicated, very small amounts of solutions (1 c.c. or less) should be taken. This advice is given, partly to secure saving of material, but chiefly to avoid the waste of time which working with large quantities always entails.

To obtain the necessary **chemical substances**, do not carry the bottles from the side-shelf to the desk. Bring a clean test-tube for liquids and a watch glass for solids. For the latter a piece of the paper provided near the side-shelf may also be used. When too much of any reagent has been taken do not return it to the bottle.

The **chemicals** are divided into three sets, each arranged **alphabetically** according to the scientific names. The first set consists of solids in small bottles, the second of the solids which are needed in greater quantities in large bottle, the third of liquids. The bottles and their places are numbered consecutively to facilitate accurate replacement, and scrupulous care must be taken not to disarrange them. Read the labels attentively, as there are frequently several kinds of the same substance (*e. g.*, pure and commercial, dilute, concentrated, and normal). For some experiments solutions of special concentrations have to be made by the student.

All **materials** are supplied through the storeroom service. Do not therefore take side-shelf bottles, when found empty, to the instructor, but to the storekeeper for re-filling.

The **nine bottles on the desk** contain aqueous solutions of sodium hydroxide, sodium carbonate, and ammonium hydroxide, which are not to be found on the side-shelves, and sulphuric, hydrochloric, and nitric acids in concentrated and dilute form. These acids are all commercial. The corresponding pure concentrated and dilute acids will be found on the side-shelf and are to be used only when the outline so directs.

All students must work independently except where cooperation of two students is expressly directed.



## CHAPTER I.

### APPARATUS.

#### 1. INSTRUCTIONS.

*a.* Read the general instructions preceding this chapter very carefully, and do not fail to observe them.

*b.* The number of blast-lamps and balances being limited, the whole class cannot perform the experiments in this chapter simultaneously in the order given. Two students from the group under each assistant, as soon as they have checked their list of apparatus, will be sent to the blast-lamp table to carry out **2** under the direction of a special assistant. Two others will pass to **5** and **6**, returning to **2**, **3**, or **4** when these are accomplished. The remainder of each section will be sent as early as possible in pairs to take the places of those returning from glass-blowing (**2**) and weighing (**6**), and meanwhile will occupy themselves with **3**, **4**, and **5**, or work in Chap. III.

*c.* Chap. II is intended for those only whose preparation in physics is defective. It is therefore to be omitted, except in cases where personal instructions to the contrary are given.

*d.* No record of **1**, **2**, and **3** is required in the note-book.

#### 2. GLASS-BLOWING [Instructions and notes below].

*a.* Cut a small piece off the wide glass tubing.

*b.* Make a test-tube of soft glass.

*c.* Make a test-tube of hard glass.

*d.* Connect two pieces of narrow glass tubing to make a longer piece.

*Notes.*—Always round off the edges of glass tubing by softening in the Bunsen flame. In the case of test-tubes, and other tubes of wide bore in which corks are to be inserted, use a pointed piece of charcoal or a file for spreading the mouth. Always distend softened parts by blowing and then cover with soot in the luminous flame before allowing finally to cool, otherwise cracks will appear.

In bending glass tubes, always use an ordinary flat luminous flame, and hold the tubing lengthwise in the flame. Never employ the Bunsen flame. Discover the reason for this injunction by bending a tube in the Bunsen flame and comparing the result with the bend made in the proper way (?). Do not bend while the tube is in the flame, but after removal (why?).

**3. CONSTRUCTION OF A WASH BOTTLE.** Select a good cork and soften it by means of the cork press. Bore two holes with a cork borer [See note below] and smooth them with a file. Prepare two glass tubes as in Fig. 1 [See note on bending under 2, *d* above],

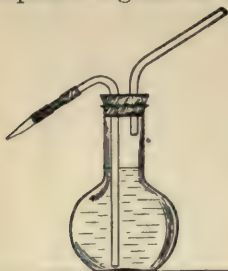


Fig. 1.

round their edges, and insert them. Make the nozzle by softening a piece of glass tubing in the Bunsen flame, drawing it to capillary dimensions, and cutting. Connect it by means of a short piece of rubber tubing [Storeroom]. Test

the apparatus to see that the joints are air-tight [Instructions]. Fill the bottle with distilled water. (Distilled water is used for nearly all experiments, and for rinsing glassware.)

*Notes.*—The cork borer is made of brass, and the edge is easily turned. Form the habit of examining the edge and freshening it by cautious application of a file [Instructions] before use. Do not hold the cork against the table while boring, as the edge of the tool may be ruined. Hold the cork in the hand and bore from the narrow end with care, exactly parallel to the axis. If the cork and borer are rotated round their axes and the edge is fresh, very little force will be required.

To avoid waste of corks, and of time in boring fresh ones, the thermometer, the stems of the dropping-funnel and thistle-tube, and the ordinary glass tubing furnished are all of the same diameter.

#### 4. BUNSEN BURNER.

*a.* Notice the effect of opening and closing the holes at the bottom of the tube. What is the proximate cause of the difference in the flames? In using the burner, always adjust the ring so as to get a noiseless, non-luminous flame.

*b.* Notice the structure of each flame. Determine which parts are relatively hotter and which cooler by placing a match and a piece of platinum wire across the flame in various parts and by causing the flame to impinge on a sheet of paper spread flat on the table. Make sketches showing the real form of the flame. Where would you hold an object in the non-luminous flame in order to get the greatest heating effect? Which region will be deficient in oxygen and which will have excess? Name those regions [R].







c. Fuse the end of a piece of glass rod (75 mm. long) in the blast-lamp and insert a short piece of platinum wire [Storeroom]. Make a bead of borax [See note below] on the straight wire (make no loop at the end), using the non-luminous flame. Observe the behavior of the borax and explain [R]. The borax is picked up by touching it with the heated wire. The bead must be small to avoid its dropping off.

d. Dissolve a speck of manganese dioxide in the bead by heating in the oxidizing part of the flame, and observe the color of the bead. If the bead is opaque, too much of the dioxide has been taken: throw the molten bead off and start again.

e. Heat this bead in the reducing part of the flame(?). To get the best result, lower the flame until it is about 6 cm. high, close the holes until a speck of luminosity appears at the apex of the inner cone, and hold the bead steadily in that spot. Before withdrawing the bead, lower it into the gas in the inner cone to cool.

f. In the oxidizing part of the flame again(?).

*Notes.*—There are six bottles on your desk, containing three acids in dilute and concentrated form. These are not pure, and should be used wherever the employment of pure acids [Side-shelf] is not explicitly enjoined. The three other bottles contain solutions of sodium carbonate, and sodium and ammonium hydroxides, which are not to be found on the side-shelf.

The chemicals upon the side-shelf are arranged alphabetically, according to the scientific names, in three sets: first, solids in small bottles; second, solids in large bottles; third, solutions and liquids. The whole side-shelf outfit is numbered consecutively, to secure easy return of each bottle to its own place. For example, borax is in bottle No. 117, labeled *sodium tetraborate*. Do not remove the bottles to your desk. Fetch the material in a watch glass, piece of paper, or clean test-tube.

## 5. MEASURING VESSELS.

a. Fit a burette with a short piece of rubber tubing and nozzle (Fig. 2). The exit may be closed either by means of a pinch clamp or by placing a small piece of glass rod in the middle of the rubber tube to choke the bore. In the latter case, pinching the tube surrounding the rod will permit the liquid to flow at any desired rate from the nozzle.

b. Fill the burette with distilled water, taking care that the air below the clamp and in the nozzle is completely expelled. Read the height of the water in the burette, observing the lower side of the meniscus and estimating tenths of a division.

Measure 10 c.c. of distilled water into each of two test-

tubes, the first with a graduated cylinder, the other with the burette. Which gives the more accurate measurement, and why? Sketch one test-tube with contents full size.

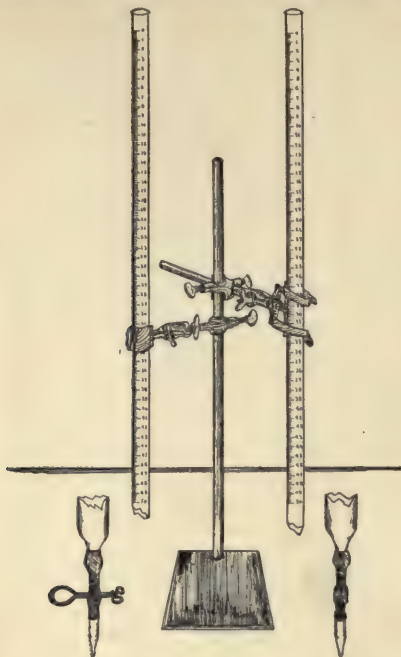


Fig. 2.

c. Measure by means of the cylinder the volumes of water your flasks and beakers hold, and record the figures. Fill them to a convenient height for use, and not to the brim.

#### 6. USE OF THE SIMPLE BALANCE [Instructions; Quant.].

a. Allow the beam of the balance to swing, and observe whether the pointer makes equal excursions on each side of the zero point. If it does not, correct the defect by placing small pieces of paper in one pan. In weighing any object, weights are added in the other pan until the vibrations of the pointer on each side are equal (not by bringing the beam to rest!).

Place a 10 g. weight in each pan, equalize the vibrations as above, add the .01 g. weight to the right-hand pan, and







find the reading about which the pointer now oscillates. This gives the deflection for .01 g. and may be used for estimating weights less than .01 g.

*Notes.*—Great care must be taken not to injure the balance or the weights. The pans of the former must be let down every time weights or other objects are added or removed. Objects to be placed on the pans must be carefully cleaned and dried. Solids to be weighed must be placed on a piece of glazed paper, or a watch glass, never on the pan directly. The weights must be lifted by means of the forceps, not by the hand. If the weights are touched by the hand, they oxidize rapidly and become inexact. In reckoning, count first by the places vacant in the box and check by counting the weights themselves. This will enable you to avoid the commonest error in weighing. Finally, record the weights in the note-book or laboratory outline, and never on loose sheets of paper. Loss of the latter, which is almost sure to occur, destroys the whole experiment, which may have occupied hours of time.

b. Ascertain the weight of a small dry beaker. Measure into it a quantity of water (about 10 c.c.) from the burette and weigh again [See note below].

In making any one of the readings of the burette, endeavor, by altering the level of the eye, to estimate what error might unconsciously be made. Note this in c.c. and express it in per cent. of the volume being measured when the reading was made.

*Note.*—Wherever in this outline instructions like the above are given, do not attempt to take the exact amount specified, but ascertain exactly how much has been taken. A quantity is indicated because one approximating that named will be most convenient for the apparatus and for securing the object in view.

## CHAPTER II.

### PHYSICAL PROPERTIES.

#### 1. SPECIFIC GRAVITY [Quant.].

*a.* Calculate from the above result (Chap. I, 6, *b*) the specific gravity of water (weight of 1 c.c.). Criticise the result. Repeat the measurement and calculation with the solution of sodium carbonate on your desk, and with chloroform. Dry the burette before filling with chloroform. Be careful not to let these or any liquids reach the pan of the balance.

*b.* Suspend a piece of thread from the hook above the pan of the balance and adjust as above until the pointer makes equal excursions on each side of the zero point. Tie to the thread a short piece of thick glass rod and weigh first in air, then in water. Calculate the specific gravity of glass.

*Note.*—Use the shelf of your pneumatic trough as a bridge on which to support the beaker of water over the pan.

*c.* Make a mark with your file in the middle of the neck of a flask holding about 25 c.c., fill with distilled water to the mark, and weigh. Weigh some small pieces of sulphur (about 2 g.), put them into the flask, bring the level of the water to the mark again, and weigh once more. Calculate the specific gravity of sulphur.

#### 2. CHARLES'S LAW [Quant.].

Take a dry flask [Instructions] of 300 c.c. capacity and fit it with a rubber stopper, short pieces of glass and rubber tubing, and clip, as in Fig. 3. Find the weight of the flask as thus fitted (*w*). Make a mark on the neck of the flask at the bottom of the stopper, and always insert the latter the same distance. Place the flask vertically in a pot of boiling water so that it is immersed completely, holding it in position by means of a universal clamp, and open the clip. After several minutes close the clip, remove the flask, invert it in a pneumatic trough filled with water,

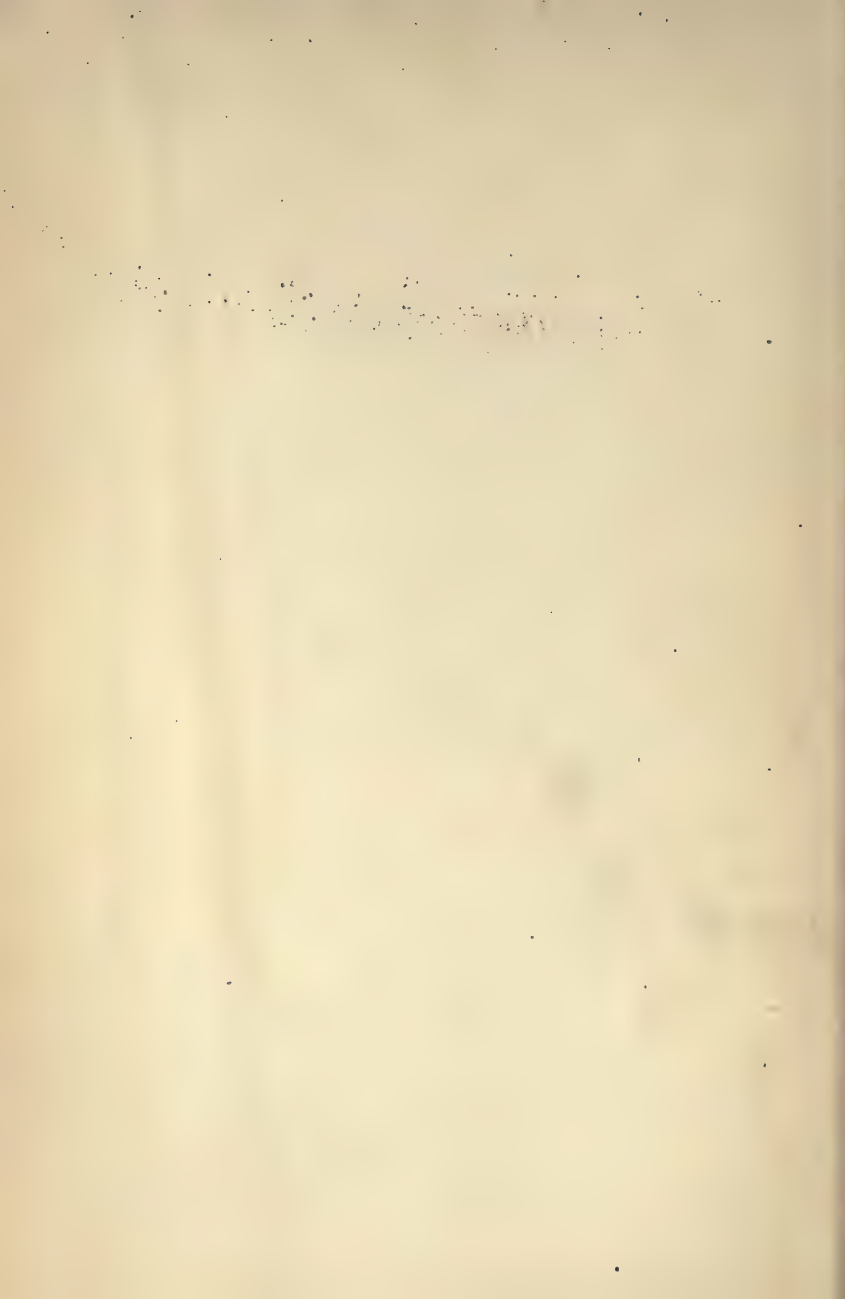


Fig. 3.

open the clip to allow the water to enter, equalize the level inside and out, close the clip, dry the outside of the flask carefully and weigh (*a*). Fill the flask and tube completely with water and weigh again (*b*). Take the temperature of the water in the trough (*t*), and of the boiling water (*t*<sub>1</sub>).







Subtracting the weight of the flask ( $w$ ) from ( $a$ ) gives the volume of water which entered (1 gr. = 1 c.c.). Subtracting the weight of the flask from ( $b$ ) gives the total volume of the flask. Subtracting these two differences, we get the volume of air which partially filled the flask at  $t^\circ$ , and completely at  $t_1^\circ$ . The difference between  $t^\circ$  and  $t_1^\circ$  gives the change in temperature. Calculate the expansion (in c.c.) of 1 c.c. heated from  $t^\circ$  to  $t^\circ + 1^\circ$ . Express this as a common fraction. What is the theoretical result calculated from Charles's law?

The result is the same whatever gas or mixture of gases is taken. (Exceptions, R?)

### 3. BOYLE'S LAW [Quant.].

Take a T tube [Temp. order] and connect one of the short arms with the apparatus used in the last experiment. (The flask must be dry as before.) Close the other short limb with a piece of rubber tubing and another clip. Immerse the long (40 cm.) limb in a beaker of mercury. Open both clips and exhaust the air as far as possible by sucking. Close the clips and measure the height of the mercury in the tube. Disconnect the T tube, leaving the clip on the flask closed. Open this under water, equalize the levels, close the clip, and weigh. Ascertain the height of the barometer ( $p$ ).

Subtracting the weight of the flask (known) from the above weight gives the volume of water which entered. Subtracting this volume from the known total volume ( $d$ ), we get the volume ( $c$ ) of the air which partially filled the flask at atmospheric pressure ( $p$ ) and completely at the reduced pressure ( $p'$ ). Get ( $p'$ ) by subtracting the height of the mercury from ( $p$ ).

Boyle's law states that  $c:d::p':p$ . Calculate ( $d$ ) from ( $c$ ), ( $p'$ ), and ( $p$ ) by this formula and compare with the value of ( $d$ ) observed.

## CHAPTER III.

### CHARACTERISTICS OF CHEMICAL CHANGE.

#### 1. QUALITATIVE STUDY OF CHEMICAL CHANGE.

*a.* Fit up an aspirator, attach it to a piece of hard glass tubing 25-30 cm. long, close the other extremity of this with a stopper fitted with glass tube, rubber tube, and clamp, and provide the other tubes and beaker all as shown in Fig. 4. The stopper of the aspirator should be of rubber. Support

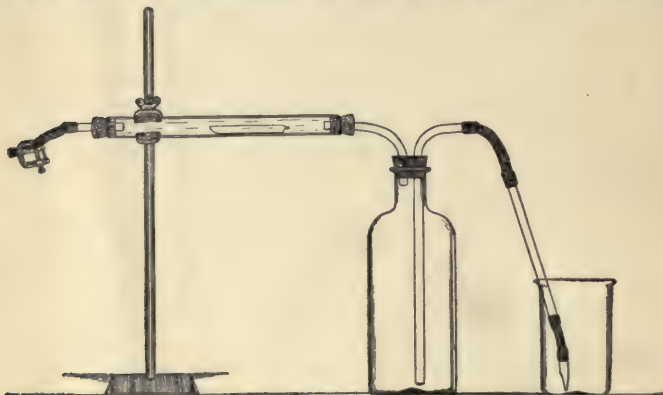


Fig. 4.

the hard glass tube at its outer end by means of a clamp attached to the iron stand. Test the apparatus and see that it is perfectly air-tight before proceeding further. To do this, place an inch or two of water in the bottle, close the apparatus, and blow a little air into the bottle through the overflow tube. If now the water remains steadily at an elevated point in the long tube there is no leak; if it recedes, the joints must be re-examined and made tight. Fill the porcelain boat with powdered iron, which should be dried previously by heating it gently in a test-tube, and, using the rough scales, ascertain the gross weight of boat and iron. [This experiment is not "quantitative."] Examine the physical properties of the iron and note the results. Place the boat in the middle of the tube. Fill the aspirator to the neck with water and put the







tubes in position, without, however, finally inserting the stopper. Displace the air in the hard glass tube with a gentle stream of oxygen from the cylinder of compressed oxygen, and then press the stopper home and pass in more oxygen until half of the water has been driven over into the large beaker placed to receive it. Turn off the oxygen and close the spring clamp behind the hard glass tube. The object of this whole operation is to expel nearly all the air and to fill the apparatus with a volume of confined oxygen. Any subsequent change in this volume of oxygen will be accompanied by the entrance or exit of water through the rubber tube and nozzle. To record the amount of oxygen at starting, leave the overflow beaker full of water, move it up or down until the levels of the water in the bottle and beaker are the same (why?), and stick a small strip of wet paper on the outside of the bottle to mark the height of the water.

Now heat the iron, at first gently, and then strongly [See note] with the Bunsen flame, noticing all that happens. If the volume of the oxygen changes, continue the operation till no further change occurs. Finally, when the tube has cooled, mark the level of the water again in the same manner as before and then weigh the boat and contents, and examine the latter. Which of the characteristics of chemical change were observable?

*Note.*—A piece of wire gauze bent in cylindrical form and placed round the part of the hard glass tube which is to be heated will diminish the risk of cracking and may be used in this and all subsequent experiments of the same nature.

*b.* Gunpowder is made from niter (potassium nitrate), roll sulphur, and charcoal. Bring specimens of these substances from the side-shelf on watch glasses, and examine them as regards properties which could be used for separation and recognition [R., Lect.]. The solubility of each in water and carbon disulphide will be most useful for the former. Examine some gunpowder, from the side-shelf. Does it resemble the constituents physically? Devise a method of ascertaining whether it is still a mixture of these substances, or has changed chemically during manufacture. Try your method (?).

*Notes.*—Pour away all ill-smelling substances, like carbon disulphide, in the sink in the hood and not in the ordinary sinks or jars.

In filtering, always cut the paper to circular form with scissors and, to avoid loss of any of the liquid, use as small a paper and funnel as will serve the purpose. Never allow the paper to project above the edge of the funnel. It should not come within less than one-quarter of an inch of the top of the latter (*cf.* Fig. 13).

**2. LAW OF DEFINITE PROPORTIONS** [Quant., *i. e.*, use finer balance and weigh to nearest cgm.].

*a.* Prepare two burettes and two evaporating-dishes. Weigh an evaporating-dish. Dilute 25 c.c. of pure [Side-shelf] concentrated hydrochloric acid [See note below] with an equal volume of water, and fill a burette with it. Fill the second burette with ammonium hydroxide solution [On desk]. From the first burette run 5 c.c. of the acid into the dish and add litmus solution until the liquid shows a distinct pink tinge.



Fig. 5.

Read the burette containing the ammonium hydroxide and run the solution into the acid, drop by drop (stirring constantly), until the exact point is reached at which the tint of the litmus is halfway between red and blue. Evaporate [Hood] the solution to complete dryness on the steam bath, cool, and

find the weight of the ammonium chloride produced. When, as in this case, no disagreeable fumes are given off, it is better to use a beaker of boiling water on the desk instead of the steam bath. After weighing, heat once more for half an hour and weigh again to ascertain whether the residue was completely dry. This precaution is taken in all experiments of this kind. The naked flame may not be used, as the ammonium chloride is volatile.

Weigh the second evaporating-dish, take 5 c.c. of hydrochloric acid as before, and add twice the volume of ammonium hydroxide used in the previous experiment. Evaporate [Hood] and weigh as before.

Compare the amounts of ammonium chloride found in the two experiments and interpret the result.

*b.* Carefully weigh an evaporating-dish and weigh out into it about 1 g. [See note, p. 3, 6, *b*] of sodium hydrogen carbonate. Dissolve in pure dilute hydrochloric acid, adding little by little and covering with a watch glass between successive additions to avoid loss by spirting. When the solid has wholly dissolved, wash the watch glass over the dish, and evaporate [Hood] the contents of the latter on the steam bath, or on a beaker of boiling water. Allow the dish to cool, and weigh. To make sure that the drying was complete, heat for half an hour or more and weigh again. Calculate the ratio by weight of the carbonate taken to the salt produced, 1 : *x*.

Repeat the experiment, using about two grams of the carbonate, and find the ratio. Compare the ratios and interpret.

A small flask is very convenient for the action of the acid on the carbonate, and the contents can afterward be rinsed into an evaporating-dish.







## CHAPTER IV.

### OXYGEN.

1. SOURCES. Heat small quantities of barium peroxide, lead dioxide, potassium dichromate, and manganese dioxide (dry this before use by heating it for a few minutes in a porcelain crucible) separately in a hard glass test-tube. Observe whether any gas is given off, and apply the test of the glowing splinter of wood [See note]. If the Bunsen flame proves inadequate, try the blast-lamp. Describe the residues.



Fig. 6.

*Notes.*—Use the clamp and ring-stand to support the tube, or grasp it by means of a strip of folded paper (Fig. 6). In either case it must be kept in a horizontal position, otherwise condensed moisture may run down and cause it to crack.

Use a long splinter of wood [Side-shelf], not a match (why?).

Clean the test-tube carefully with hot nitric acid and dry it after each experiment.

Beginning with this chapter include in your notes equations for all the chemical changes you observe. When no change is observed, do not attempt to give an equation. In the present experiments the formulæ of the materials used will have to be sought in the text-book. The formulæ of the products will also be sought in the book after the appearance of the product and the evolution or non-evolution of oxygen have been noted and an indication of what to seek for has thus been obtained.

Commercial manganese dioxide sometimes contains charcoal, and it is therefore dangerous to use it for mixing with potassium chlorate in making oxygen in 2 without first heating a little alone. Signs of glowing indicate the presence of a combustible substance.



Fig. 7.

2. PREPARATION. Mix on paper about 5 g. of potassium chlorate and 3 g. of powdered, dried manganese dioxide. Put the mixture in a hard glass test-tube closed with a cork and fitted with a delivery tube (Fig. 7). Test the apparatus to see that it is air-tight. Clamp the test-tube on the stand. Heat carefully, so as not to cause too violent an evolution of gas, and collect in four bottles over water. Remove the deliv-

ery tube from the water as soon as the bottles are full (why?). When the test-tube is cold, the contents may be cleaned out by allowing them to soak in cold water.

### 3. PROPERTIES.

*a.* Lower a little ignited sulphur in a deflagrating spoon into one bottle (?). Remove the spoon, add a little water, close with the hand, and shake (?). Test the water with blue litmus paper or solution [R].

*b.* Lower a very small piece of burning phosphorus in the spoon into the second bottle in the same way (?). [Instructions: Phosphorus must always be cut under water and handled with forceps. Great care must be taken not to touch it with the hand, as it catches fire easily, and causes very severe burns. Red phosphorus is safer, and should be substituted here if available.]

Proceed as in *a* (?). Test with blue litmus [R].

*c.* Lower a splinter of glowing charcoal into the third bottle, holding it in the tongs or wrapping the end of a piece of copper wire round it (?). Treat as before (?). Test with lime water [R].

### 4. CHEMICAL ACTIONS INVOLVED IN THE PREPARATION OF OXYGEN [Quant., balance].

*a.* Devise a way of separating the substances left in the tube used in 2 and of ascertaining whether the manganese dioxide has been altered. Consult the instructor in regard to the details of your plan before proceeding to execute it. Weigh the manganese dioxide. Do not use any delivery tube or collect the oxygen. Recover the potassium chloride and compare its appearance with that of the chlorate. Test solutions [Side-shelf] of each with silver nitrate solution and explain the result [R].

5. SLOW OXIDATION OF METALS. Devise a way of showing that air loses a part of its substance when moist iron filings rust, and try it. Submit your arrangement to the instructor for criticism.

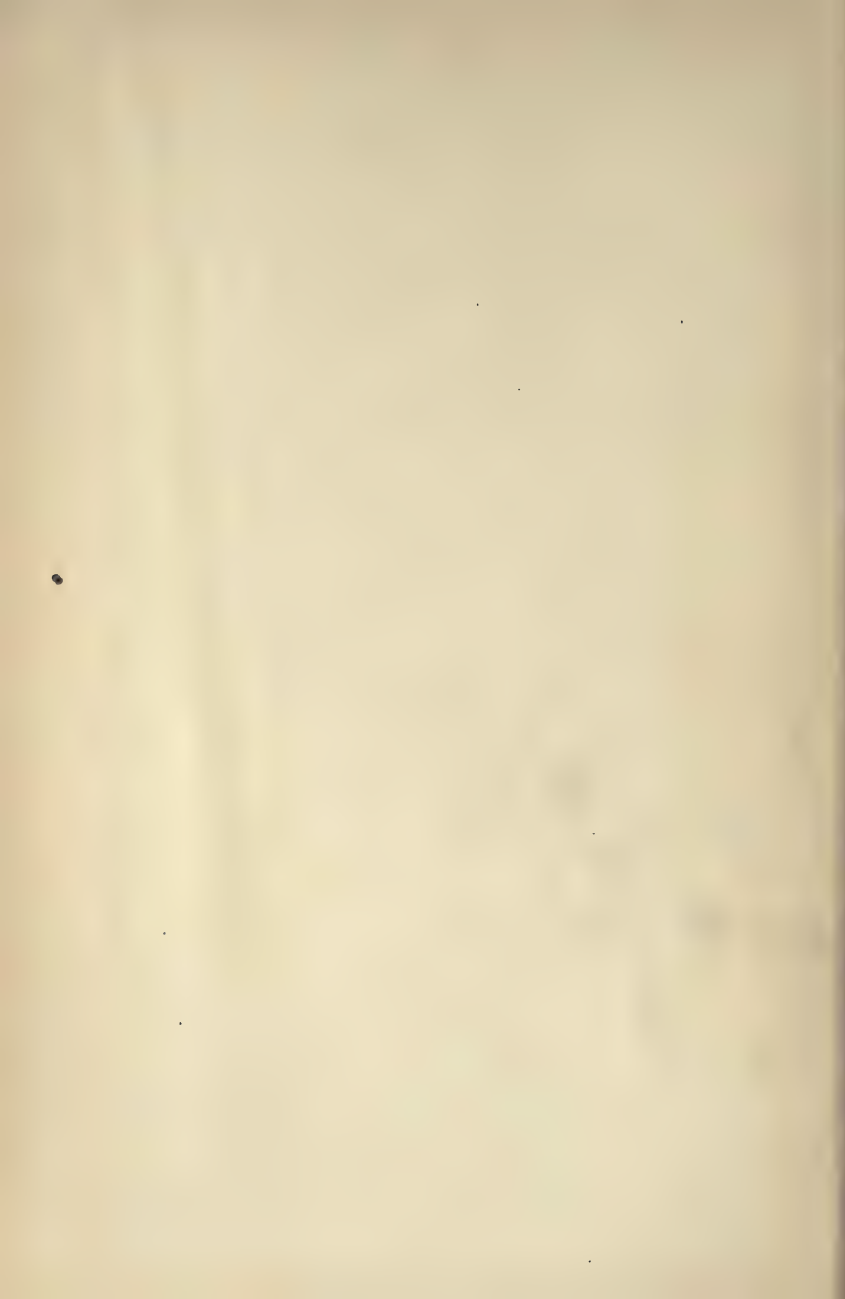
### 6. WEIGHT OF A LITER OF OXYGEN, USING ASPIRATOR [Quant.].

*a.* Powder some potassium chlorate, and dry it on a watch glass on the radiator or high above a small Bunsen flame. Construct an aspirator (Fig. 8), using the 1-liter bottle, and connect it with a hard glass test-tube.<sup>1</sup> Fit a nozzle to

<sup>1</sup>Really infusible test-tubes often cannot be obtained. They have to be made to order. Test-tubes made by the students themselves from good combustion tubing are more trustworthy.







the rubber tube. Test the apparatus to see that all the joints are air-tight [Instructions: Place some water in the bottle, blow a few bubbles of air into the apparatus through the syphon, and observe whether the water remains permanently elevated in the vertical tube]. Fill the bottle with water so that the short tube is not immersed. Fill the rubber tube and nozzle completely with water and close the clamp. Weigh the hard glass tube, put into it about 1 gr. of the chlorate, weigh again, and connect with the bottle. The weight of



Fig. 8.

chlorate taken, whatever it may be, must be known exactly: it should not much exceed 1 gr. Allow the rubber tube to hang to the bottom of a beaker (400 c.c.) containing some water. Open the clamp and raise the beaker till the levels of the water in this and the bottle are the same, and the gaseous pressure therefore alike in both. Close the clamp again, empty the beaker, and replace it.

Open the clamp once more and decompose the salt slowly by heating, catching in the beaker the water driven over by the gas. Stop heating if the tube shows signs of softening, or when the decomposition is complete. For the purpose of this experiment (see 6, *b*) it is not necessary that the action should be carried to completion. If, when the heating is stopped, the nozzle is not under water, raise the beaker until it is well covered. Allow the whole apparatus to stand until it has reached the temperature of the air. Some water will return to the bottle by the tube during the cooling. Admission of air to the tube through the nozzle at any stage of the operations will prevent this transference, which is essential to the success of the experiment. Equalize the level of the water in both vessels by raising or lowering the beaker, and then close the clamp.

Measure the volume of water in the beaker by weighing

the vessel first with and then without the water on the laboratory scales and subtracting. It will be equal to that of the gas evolved (what of the air originally in the apparatus?). Weigh the tube once more with care, and observe the temperature and pressure of the air.

Subtract the aqueous tension at the observed temperature (see Appendix) from the barometric reading to get the true (partial) pressure of oxygen in the bottle. Reduce the volume to  $0^{\circ}$  and 760 mm. Subtracting the weight of the salt remaining from that of the chlorate gives the weight of this volume of oxygen. Calculate the weight of 1 liter and the volume of 32 gr.

Test aqueous solutions of the chloride and chlorate with silver nitrate solution and explain the result [R].

To what class of gases would the use of the aspirator be confined for purposes like the above?

*b.* If the condition of the hard glass tube from 6, *a* will permit, detach and heat it with its contents once more to drive out the last traces of oxygen. Allow it to cool and weigh it. Obtain the weights of chloride and total oxygen by difference.

Calculate the ratio of the weight of the potassium chloride to that of the oxygen, assuming the formula of the former (KCl) and its formula weight 74.5 ( $74.5 : x$ ).

Assuming the combining weight of oxygen to be 16, determine the formula of the chlorate.

Show how this gives the equation for its decomposition by heat.







## CHAPTER V.

### EQUIVALENT WEIGHTS, FORMULÆ, AND EQUATIONS.

#### 1. COMPOSITION OF CARBON DIOXIDE [Quant.].

*Note.*—Two students may with advantage carry out this experiment together.

Fit a piece of hard glass tubing, 25–30 cm. long, with perforated corks and insert at one end a short piece of glass tubing and at the other a U tube, as in Fig. 9. The inner edges of the hard glass tube should be rounded with a file or flared by use of the blast-lamp and a piece of charcoal.



Fig. 9.

Rubber stoppers will give tight joints more surely than corks. Attach to the U tube by means of a cork a short straight tube, of the diameter of a narrow test-tube, which has been drawn out so as to leave a small opening at the free end. Arrange a loop of wire with which to suspend the potash tubes from the balance. Place in the hard glass tube a plug of granular cupric oxide, or an oxidized roll of copper gauze, about 4 cm. in length. The former may be held in position by small wads of asbestos. The cupric oxide and asbestos must be dried by heating in the porcelain crucible before use. Put about 0.2 g. of pure dry sugar-charcoal [Storeroom] in a porcelain boat, weigh the boat with contents, and set it in the tube close behind the cupric oxide. Make a few c.c. of a strong (approximately 30 per cent.) solution of potassium hydroxide, fill the bend of the U tube with it, and charge the small tube beyond with fragments of solid caustic potash. Test the apparatus to see that it is air-tight [Instructions]. Finally, weigh the connected potash tubes immediately before starting the combustion, suspending them on the balance by means of a thread.

Slip the cylinder of wire gauze over the hard glass tube, connect the latter with the oxygen cylinder (or other source

of oxygen), and heat the part containing the boat and cupric oxide with two burners [Temp. order]. Turn on the oxygen with care and regulate the stream so that the carbon may burn slowly and not more than 15-20 bubbles of unused oxygen escape per minute. A more rapid stream will involve the loss of carbon dioxide. Heat the front of the boat first and let the glow, caused by the combustion, travel along. The burning will take 30-45 minutes. Continue the stream of oxygen for 4-5 minutes after the carbon is completely burned (why?), then disconnect the potash apparatus and weigh it. A more accurate result is obtained by finally displacing the oxygen by air (why?). After the tube has cooled, weigh the boat with any ash it may contain. Return the cupric oxide to the bottle.

The loss in weight of the boat gives the amount of carbon; the gain in weight of the potash apparatus, the amount of carbon dioxide. The difference of these two gives the oxygen.

Calculate the percentage composition of carbon dioxide and the weight of carbon combining with 8 parts of oxygen. This gives the equivalent weight of carbon.

Assuming the atomic weights of carbon and oxygen to be 12 and 16 respectively, calculate the formula of carbon dioxide from the percentage composition.

Make the equation representing the action.

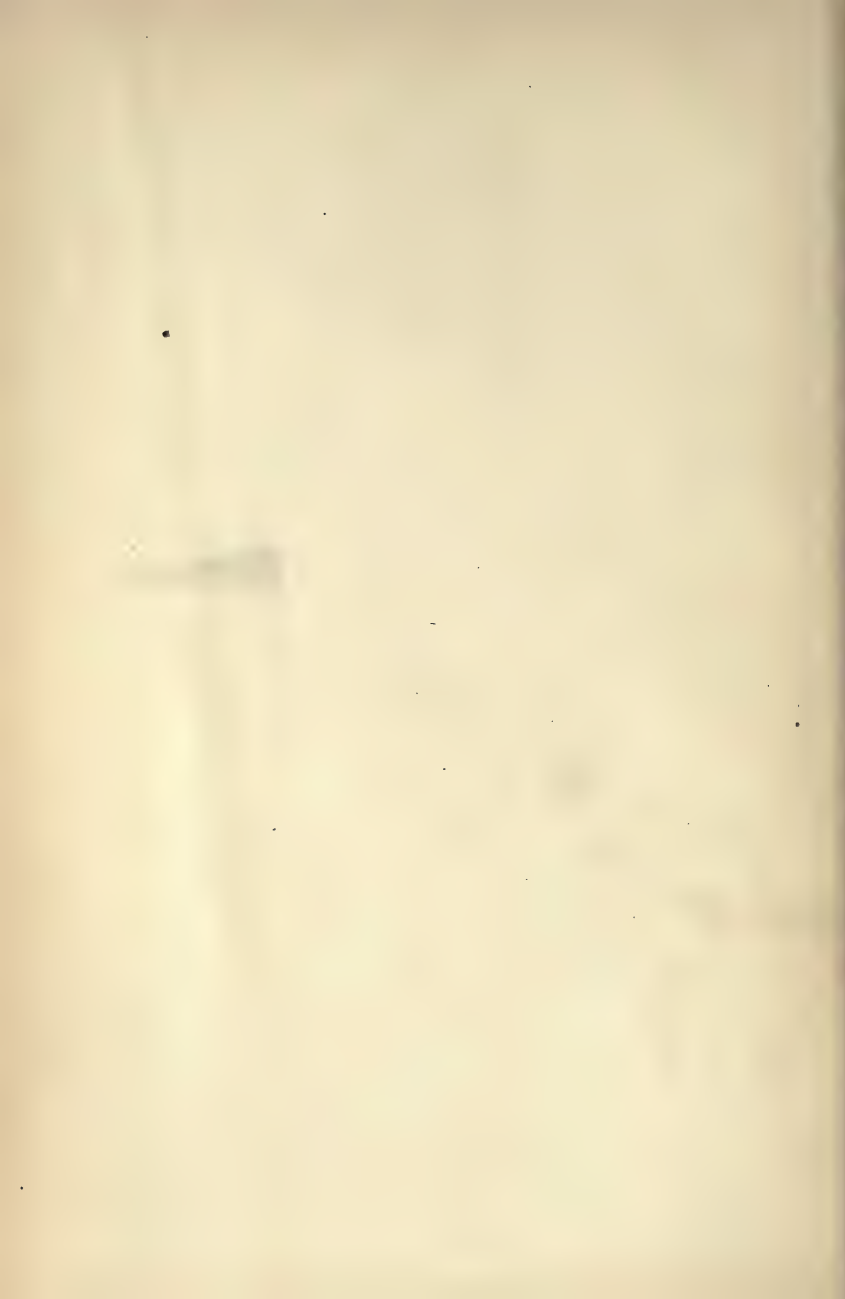
What would be the formula of the compound if the symbols represented the equivalent weights?

## 2. COMPOSITION OF AN OXIDE OF A METAL [Quant.].

On account of the difficulties attending the making, the collecting, or the weighing of most oxides formed by direct union, the following indirect method is suggested. It consists in converting a known weight of a metal into nitrate by the action of nitric acid and obtaining the oxide by ignition of this salt (attempt no equations for these actions).

a. Weigh an evaporating-dish of medium size, place in it about 0.5 g. (6 inches) of pure iron wire [Storeroom], and weigh again. Cover the dish with a watch glass, convex side downward, and add 5 c.c. of pure dilute nitric acid. Set the dish, covered, on the steam bath until the iron has dissolved. Then rinse the cover-glass carefully into the dish and evaporate the solution to dryness on the steam bath or on a beaker of boiling water [Hood]. When residue (what is it?) is dry, place the dish on a wire gauze and heat carefully with a burner held in the hand as long as any red fumes are given off. Allow the dish and contents (?) to cool and weigh them. To make sure that the decomposition was complete, heat once







more, cool, and weigh again. This precaution is always necessary in experiments of this nature.

Assuming the equivalent weight of oxygen as 8, calculate that of the iron in the oxide.

Assuming the combining weights of iron and oxygen to be 56 and 16 respectively, determine the formula of the compound.

b. Pure zinc (about 0.5 g.) may be used instead of iron. The manipulation is the same as in 2, a. The residue from evaporation is a syrup-like body, however, which cannot be dried. Extra caution must be used in heating this to avoid loss by spirting.

Answer the same questions as in 2, a.

### 3. EQUIVALENT WEIGHT OF A METAL BY DISPLACING HYDROGEN [Quant.].

a. First fill the pneumatic trough and 1-liter bottle with water so that the latter may acquire the temperature of the room. Boil about half a liter of water and allow it to cool. Fit a 100 c.c. flask somewhat as in Fig. 10, using in place of the thistle-tube a dropping-funnel. To carry a doubly bored cork the mouth of the flask must be rather wide. A larger flask than 100 c.c. must not be used on account of the waste of acid it would entail. The stem of the dropping-funnel must reach to the bottom of the flask, and the inner end of the other tube must be flush with the bottom of the cork. Attach a rubber or glass delivery tube and see that the apparatus is air-tight. Weigh a piece of chemically pure zinc, taking about 2 g. Without detaching your platinum wire from the glass rod, wrap it tightly round the zinc (why?) and allow the whole to slide gently into the flask. Fill the apparatus completely, from the top of the stem of the funnel to the tip of the delivery tube, with part of the boiled water. Close the stop-cock when the bulb has almost emptied itself. Invert the 1-liter bottle, filled with water, on the shelf of the pneumatic trough, and put the delivery tube in position.

Half fill the globe of the funnel with pure concentrated hydrochloric acid [Side-shelf] and admit this to the flask, a little at a time, in such a way that a steady, but not too violent, action takes place. When the metal is entirely dissolved, drive all the gas over into the bottle by pouring boiled water once more through the funnel (be careful that no air is carried with the water).

When the gas has acquired the temperature of the water and room, lower the bottle until the level of the water outside and inside is the same. Close the bottle while it is in this

position with a cork and remove it from the trough. Weigh the bottle as it stands on the laboratory scales, and also completely filled with water, and subtract, to find the volume of the gas. Take the temperature and barometric pressure. Subtract from the latter the aqueous tension (Appendix). Find by calculation (1 liter weighs .09 g. at  $0^{\circ}$  and 760 mm.) the weight of the hydrogen obtained. Calculate the equivalent weight of zinc, *i. e.*, the weight of the metal which displaces 1.0076 g. of hydrogen. Wash the trough carefully until it is absolutely free from acid and put it away in an inverted position to avoid rusting.

*b.* The experiment may be performed with magnesium (about 0.7 g. = 65 cm. of No. 23 wire), iron (about 1.5 g.), or aluminium (about 0.8 g.) in place of zinc.

In the absence of a dropping-funnel, a substitute may be made by connecting a funnel with a straight tube by means of a rubber joint closed with a pinch clamp. Or, the aspirator (Fig. 8) may be used here, the metal (half the above quantities), water, and a smaller tube containing the acid being placed in a test-tube, and the mixing being effected by inclining the bottle after the apparatus is connected.

#### 4. INTER-EQUIVALENCE OF EQUIVALENT WEIGHTS [Law of Reciprocal Proportions].

If **2, b** was performed, compare the quantity of zinc which combined with 8 g. of oxygen with that which in **3, a** was found to displace 1.0076 g. of hydrogen. If they are identical, these two quantities of oxygen and hydrogen may combine with one another. What substance has precisely this composition?

#### 5. COMPOSITION OF ZINC CHLORIDE [Quant.]. (From Torrey's *Elementary Studies*.)

Weigh an evaporating-dish of medium size and place in it about 2 g. of pure zinc. Add a little diluted (1:2), pure, concentrated hydrochloric acid [Side-shelf] and cover with a watch glass, convex side downward. If the action is very slow, the tip of a platinum wire may be placed in contact with the zinc. Maintain a brisk action by further additions of concentrated hydrochloric acid in very small amounts at a time. Final excess of the acid should be avoided, as time will be lost in the subsequent evaporation. When the metal is completely dissolved, rinse the cover-glass and platinum wire carefully into the porcelain dish and remove them. Allow the solution to evaporate as far as possible on the steam





bath or on a beaker of boiling water [Hood]. Now place the dish on the ring-stand and, using a small Bunsen flame, allow the syrup-like solution to evaporate slowly to dryness. Then heat the white mass to the point at which it has completely melted and no further. The best way to achieve this with the minimum rise in temperature is to let the Bunsen flame play on the surface from above. Overheating must be avoided, because the product is volatile at high temperatures. The moment the dish has so far cooled that the hand can be borne upon the bottom, wipe the dish carefully and weigh it. The substance absorbs moisture greedily from the atmosphere, hence expedition is required in cooling and weighing if accurate results are to be obtained. To insure accuracy, the melting, cooling, and weighing should be repeated, and the lower result taken as correct.

Using the equivalent weight of zinc obtained in 3, *a*, calculate the equivalent weight of chlorine.

Assuming the combining weights of zinc and chlorine to be 65 and 35.5 respectively, determine the formula of zinc chloride.

Using the result of 3, *a*, find how many combining weights of hydrogen are displaced by 65 parts, the combining weight, of zinc. What is the valence of zinc?

Express the whole action of hydrochloric acid on zinc in symbols by making the equation in accordance with these conclusions.

## 6. MULTIPLE PROPORTIONS [Quant.].

*a*. [Two students working together.] Fit a hard glass tube, 25-30 cm. long, with corks, through which pass short pieces of narrow glass tubing. To one of these connect a Kipp's apparatus for generating hydrogen and a gas washing bottle (shown in Fig. 12) one-half full of concentrated sulphuric acid. Make sure that the apparatus is air-tight. Dry some powdered cupric oxide by heating it in a hard glass test-tube. Weigh two clean, dry, porcelain boats [Temp. order] and place in one about 1 g. of the cupric oxide and weigh again. Have about 1 g. of pure cuprous oxide [Storeroom<sup>1</sup>] placed in the other and weigh likewise. Be careful in recording the weights and in handling the boats to distinguish the one from the other. Place the boats in the hard glass tube, that containing cupric oxide in front, and surround it with a piece of wire gauze.

<sup>1</sup> Pure cuprous oxide (Kahlbaum's) can be kept successfully if sealed up in small bottles which are not opened until needed.



Pass a gentle stream of gas through the apparatus until a test (?) shows that the air has all been displaced. Now heat the boats moderately, beginning with the front one. What collects in the rear of the boat? Where does it come from? When the action, which requires 10–15 minutes, is finished, allow the boats to cool in a stream of hydrogen. Weigh the boats and contents (?), taking care not to interchange them. To ascertain whether the action is complete heat the boats once more in hydrogen, cool, and weigh again.

Assuming the equivalent weight of oxygen to be 8, calculate, from your results, that of copper in each oxide. What is the ratio of the two values for copper?

Using your results, and assuming 16 and 63.6 to be the atomic weights of oxygen and copper respectively, find the formula of each oxide. Construct the equations representing the action of hydrogen.

b. If the experiment in Chap. IV, 6, b, (p. 14) was carried out, dried potassium perchlorate may be decomposed in the same way and the results compared. Simply use an open, long, hard glass test-tube, without aspirator, and drive off the oxygen slowly.

Calculate the ratio of the weight of the potassium chloride (KCl) to that of the oxygen, assuming the formula weight of the former, 74.5, as before ( $74.5 : x'$ ). Compare the value of  $x'$  with that of  $x$  in the former experiment. What are the smallest integers which will express their ratio ( $x : x'$ )?

Assuming the atomic weight of oxygen to be 16, determine from your results the formula of the perchlorate.

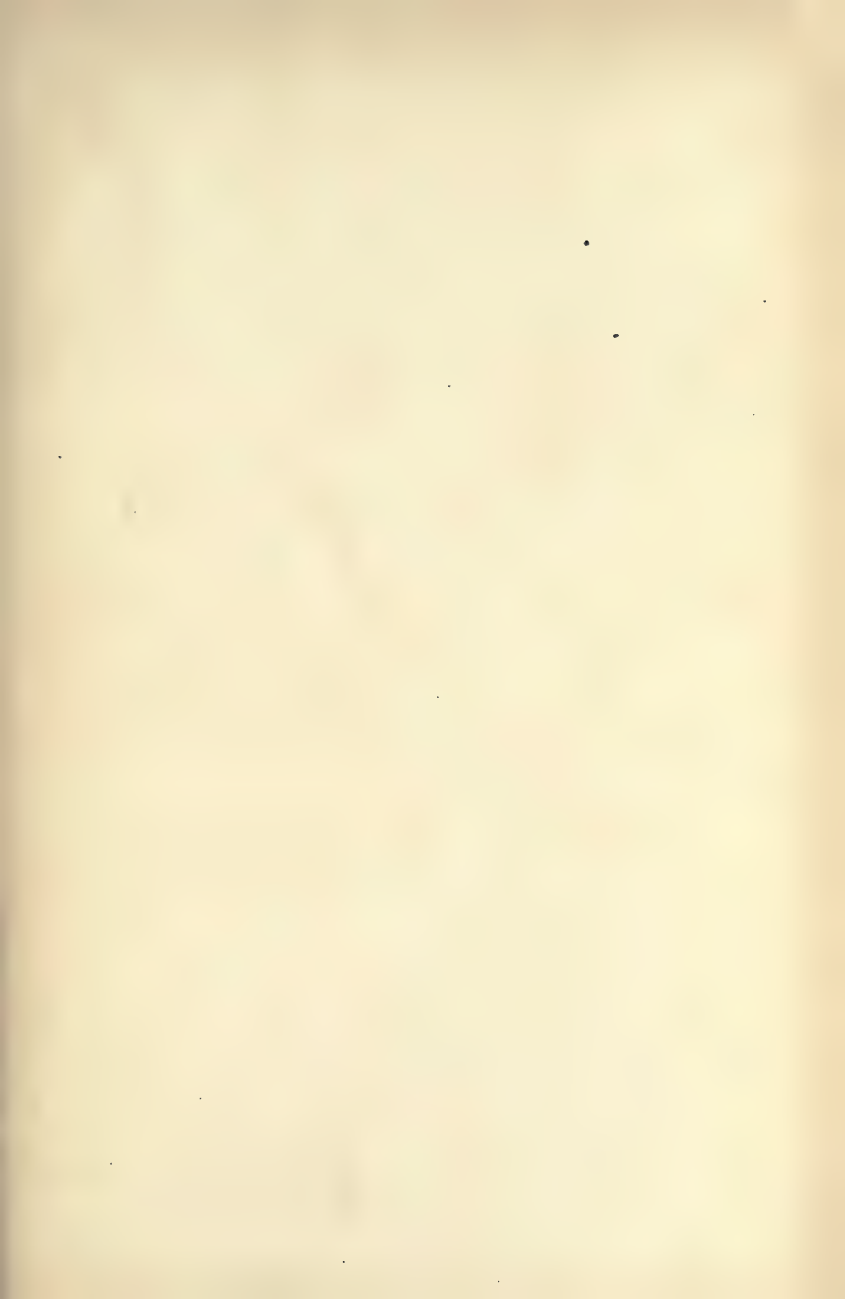
Construct the equation for the action.

c. Pure lead dioxide and pure lead monoxide may be used as in *a*, if available. The monoxide, however, absorbs carbon dioxide readily from the air, and therefore does not keep well. It is also more difficult to reduce than the dioxide. The heating in a stream of hydrogen must be repeated to constant weight.

**7. DULONG AND PETIT'S LAW.** The specific heats of the metals used in this chapter are:

Aluminium	0.214	Lead	0.031
Copper	0.095	Magnesium	0.250
Iron	0.114	Zinc	0.095

According to Dulong and Petit, if the correct atomic weight, when it has been found, is multiplied by the specific heat of the element in the solid form, the product is a number which in most cases lies between 6 and 6.8.





Take the values of such combining weights and equivalents as you have found experimentally in **2** (iron or zinc), **3** (zinc, magnesium, iron, or aluminium), **5** (zinc), **6** (copper), and multiply each by the corresponding specific heat. If the result is about 6.4, the atomic weight is the same as the equivalent weight. If not, multiply the equivalent weight by the smallest integer which will bring the final product within the limits 6 to 6.8. The integer used is the valence of the element, and the product of equivalent weight into the valence is the atomic weight.

## CHAPTER VI.

### HYDROGEN.

#### 1. INTERACTION OF METALS AND ACIDS.

a. Place a small piece of each of the metals, tin (gran.), copper (turnings), iron (filings), zinc (gran., large bottle), lead (gran.), aluminium (wire), and magnesium (wire) in separate test-tubes and add to each a little pure [Side-shelf] concentrated hydrochloric acid. (What is the nature of this liquid? [R.]) Observe each case critically, and record the results, noting the order of the metals in respect to activity. Note the effect of heating, if no action occurs in the cold. If heating seems to produce gas, remember that it may be hydrogen chloride or steam and not hydrogen. It will be necessary to use discrimination in deciding this point. After the action has ceased, filter any one of the solutions and evaporate [Hood] it to dryness on the sand bath (?).

Can you give any grounds for the belief that the hydrogen comes from the acid and not from the metal or the water?

b. Ascertain the influence of the physical state of the metal on its apparent activity by using zinc dust with the same acid.

c. Try in the same way the action of dilute sulphuric acid on copper, zinc (pure), and iron. In case little or no action takes place in the cold, try the effect of putting a platinum wire in contact with the metal (?) and, having withdrawn the wire, of adding a drop of cupric sulphate solution to the two last (?) before resorting to heating. If any effect is observed, notice where the hydrogen appears to come from, and explain [R]. When the acid has been exhausted by the action of the last metal (of which excess must be used) filter the solution while it is still warm from the action, and set it aside on the shelf of your cupboard to crystallize [Instructions and notes below].

d. Try the action of strong acetic acid on iron

e. For comparison, study the action of a little concentrated sulphuric acid on zinc (gran.) in the cold and in the heat. What products are formed?

*Notes.*—In observing an interaction a chemist first mixes the substances thoroughly by shaking. If nothing occurs, he then heats. If his eye shows evidence of the production of a gas or vapor, he finally smells the contents of the tube. Apply these







three methods of observation to *e* before drawing any conclusion.

In making the equations for the above actions all that is needed, beyond the information given above and acquired by observation, is to find the formulæ of the products. These are not here to be sought by measurement, but in the text-book [R].

## 2. INTERACTION OF METALS AND WATER.

*a.* Recall the action of sodium and potassium on water [Lecture].

*b.* [Two students working together.] Place about 20 cm. of magnesium ribbon, twisted into a knot, in the higher end of an inclined hard glass delivery tube. Fit a 250 c.c. flask, like that used in generating hydrogen (Fig. 10), and heat some water in it until a regular stream of steam issues. If the stream is unsteady, put some broken porcelain into the flask. Now heat the magnesium with another burner, connect the flask so as to pass steam over the heated metal, and collect the resulting gas over water in a bottle. When enough has been collected, or the magnesium is all oxidized, remove the delivery tube from the water, and apply a light to the gas in the bottle.

*c.* The molecule of sodium hydroxide, formed from a molecule of water in *a*, may undergo further change by heating with a fresh quantity of sodium. It is more convenient to use another metal for the purpose, however.

Prepare a hard glass test-tube and a delivery tube. Rapidly powder some sodium hydroxide (1 part), mix it with zinc-dust ( $1\frac{1}{2}$  parts), heat in the tube clamped in a horizontal position, and collect the gas over water. Ascertain whether the gas is combustible. What information about the original molecule of water do the results of *a* and *c* together give us? What is the solid product in *c* [R]?

*d.* Place about 5 g. of granulated zinc in a test-tube and let it stand, for a few seconds only, with a dilute solution of cupric sulphate (?). Pour off the solution and add some water. Take special care not to shake with air, as the copper is easily oxidized and its effect impaired. Fit the test-tube with a cork and delivery tube and boil persistently, using a small flame. After the air in the tube has been completely displaced by steam, collect the gas, which comes off very slowly, over water in a test-tube. Determine whether it is combustible. Make a general statement about zinc on the basis of 1, *a* and *c*, and 2, *d*; also about the hydrogen in water and acids.

## 3. PREPARATION OF HYDROGEN [Same apparatus is used for 4 and 5].

*a.* Put some granulated zinc [Large bottle] in a 250 c.c.

flask (Fig. 10) provided with a rubber delivery tube, add diluted hydrochloric acid, and test the issuing gas till it is found free from air. [Instructions: The gas must not be ignited at once, or the apparatus may be blown up. Samples must be caught in test-tubes, held bottom up, and the tubes must be brought

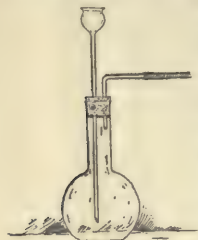


Fig. 10.

to a distant flame in the same position. If the gas contained in them, after the first explosion of the part nearest to the mouth, burns quietly, the gas is free from air.]

Ascertain whether ordinary combustibles burn in the gas. Show whether it is lighter or heavier than air. Fill a test-tube with one-third hydrogen and two-thirds air and apply a light to the mixture. Keep the apparatus in operation for use in 4 and 5.

**4. PRODUCT OF THE UNION OF HYDROGEN AND OXYGEN.** Attach a glass tube, drawn out to a capillary opening, in place of the delivery tube. Ascertain that the issuing gas is not explosive (test?). Hold a cold, dry beaker close against the jet of unlighted gas (?). If moisture is deposited (what is its source?), insert a U tube filled with chloride of calcium to dry the gas. When the gas no longer deposits moisture, set fire to it and hold the beaker over the flame (?).

Burn ordinary illuminating gas from the same nozzle and hold the beaker over the flame (?).

## 5. REDUCTION.

*a.* [Two students working together.] Fit a hard glass tube open at both ends with perforated corks and short tubes. Place aluminium oxide, ferric oxide, and minium, one at a time, in the tube near the end next to the generator, but not so near as to endanger the corks when heat is applied. Dry each oxide by heating in a porcelain crucible before use. Support the tube on the stand by means of a clamp attached close to one end, connect it with an apparatus delivering dry (see 4) hydrogen. [Use the generator made in 3, or a Kipp's apparatus, as a source of hydrogen.] Heat each oxide in a slow stream of the gas. After each time the apparatus has been opened, wait till the air has been displaced by hydrogen before heating (why? test?). Observe the effect on each oxide and whether water condenses in the tube. If no effect is noticed after the oxide has been at a red heat for three minutes, absence of action may be inferred.

Ascertain the common names of the oxides used [R]. Of







what chemical change shown in the lecture-room is the action on ferric oxide essentially a reversal? [Advanced students. What conditions permit the completion of the action in either direction? R.]

b. Mix thoroughly in a mortar equal bulks of magnesium powder and powdered calcium carbonate. Put the mixture in a test-tube (it should fill about half an inch of the tube), fix the tube in a clamp on the stand, and heat the top layer in the Bunsen flame until the reaction begins. It will go on to completion by itself. Be careful to keep the tube directed away from the face during the heating. Allow the test-tube to cool, add a little water, and then, slowly, an excess of concentrated hydrochloric acid (?). If the tube has been broken, place the contents with the acid in a beaker. The acid will dissolve any excess of calcium carbonate or magnesium with effervescence. It will also dissolve the oxides of magnesium and calcium formed by the action. The black material alone should remain undissolved. When all action has ceased, filter and wash the black residue (?) with water. After drying this on the radiator or steam bath, prove that it is carbon. This may be done by placing some of it in a dry test-tube, adding a pinch of potassium chlorate, heating in the Bunsen flame, and pouring the gas when it has cooled (close the tube with the thumb while waiting for this) into a test-tube containing lime water and shaking (?).

What is the reducing agent in this action?

## CHAPTER VII.

### WATER AND SOLUTION.

**1. PURITY OF WATER.** Place a few drops of distilled water on a clean watch glass (why not an evaporating-dish?) and evaporate on the steam bath. Do the same with ordinary water. Observe whether any stain remains on the glass. What class of impurities would leave no trace of their presence in this test?

**2. UNION WITH OXIDES.** Place a pinch of cupric oxide in a test-tube and wash it by shaking with a little distilled water and pouring off the liquid. Add more water and shake again. Test this solution with neutral litmus paper (?). At the same time test a sample of the water with the same paper and compare the tints. Repeat with barium oxide (?).

The behavior of acid-forming oxides has been examined in Chap. IV, 3 (p. 12). Recall the three cases.

### **3. HYDRATES.**

*a.* Heat some blue vitriol gently in a porcelain crucible (?). Dissolve the white powder by boiling with the minimum amount of water required to dissolve it and set the solution aside (?). What chemical action has taken place in each case?

*b.* [Quant.] Place small quantities (about 1 g.) of Glauber's salt and blue vitriol in two porcelain dishes and ascertain the gross weight in each case. Allow the dishes and contents to remain for 24 hours or more and weigh again (?) [R].

*c.* Gently warm small quantities of barium chloride, potassium nitrate, magnesium sulphate, alum, and potassium dichromate separately in dry test-tubes and notice whether they undergo any change [R].

Are all crystalline substances hydrates? Classify the substances you have examined into two groups with reference to this property.

*d.* [Quant.] Weigh out about 2 g. of crystals of gypsum in a porcelain crucible. Heat to redness until no further loss in weight occurs, and weigh the calcium sulphate remaining. Assuming the atomic weights of the elements and the formulæ of the sulphate and of water, calculate the formula of gypsum.







4. **DELIQUESCENT.** Set some potassium carbonate aside in a small beaker and examine it from day to day.

5. **SOLUTION: GASES IN LIQUIDS.**

a. Half fill a 1-liter bottle with distilled water, cork, and shake vigorously till the water is saturated with air. Take the temperature of the water and the height of the barometer.



Fig. 11.

Measure the content of a flask holding about 100 c.c. and fit it with a cork and delivery tube (Fig. 11.) Fill the whole apparatus, including the tube, completely with the prepared water, and boil, collecting the gas in an inverted test-tube.

When no more comes over, equalize the levels and mark the level in the tube with a thin rubber ring. [Cut this from your rubber tubing.] Measure the volume which the air occupied and calculate the volume of air dissolved by 100 c.c. of water at the observed temperature and pressure, correcting for the aqueous vapor present (?).

What proportion of its own volume of air has the water dissolved?

*Note.* An inverted burette partially filled with water and closed at the top may be used in place of the test-tube, if proper correction for difference in level of the water is made. Divide the height of the water by 13.6 to get the equivalent in mercury and subtract from the barometer.

b. [Quant.] Fit up a gas washing bottle and test-tube (Fig. 12), making a slight notch down the side of the cork in the latter to permit the escape of gas. Provide a short piece of wire for hanging the test-tube on the balance. Fill the bottle one-third full of water (use of this?).

Weigh the test-tube and fittings, place in it about 5 c.c. of water, weigh again, and finally support it in a beaker full of cold water. Connect a bottle of liquid sulphur dioxide [Hood] with the wash-

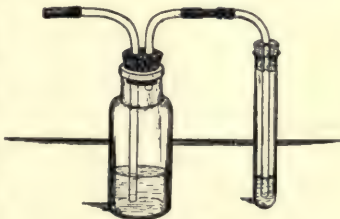


Fig. 12.

bottle and turn on a slow stream of the gas. When the water seems to be saturated, stop the gas. Disconnect, dry, and weigh the test-tube once more. Repeat the operation for a minute or so and weigh again. If there is an increase,

repeat until the weight is constant. Take the temperature ( $t$ ) of the water in the beaker and the height of the barometer ( $p$ ). Calculate the weight of sulphur dioxide dissolved by 100 c.c. of water at the observed  $t$  and  $p$ . Calculate the volume which this weight of gas would occupy at  $t$  and  $p$ . How many times its own volume of the gas has the water dissolved?

State the law (Henry's) in accordance with which the amount of the gas dissolved varies with the pressure [R].

#### 6. SOLUTION: LIQUIDS IN LIQUIDS.

*a.* Take 5 c.c. of alcohol in one test-tube and an equal volume of water in another. Add the alcohol to the water a drop or two at a time, shake after each addition, and observe whether solution takes place (?). Repeat, adding water to alcohol (?).

*b.* Repeat *a* with ether and water and with carbon disulphide and water (?), taking care to use dry test-tubes.

#### 7. SOLUTION: SOLIDS IN LIQUIDS.

*a.* Prepare a saturated solution of potassium dichromate by pulverizing the salt (about 7 g.), placing it in a flask containing 50 c.c. of water, and shaking it at intervals for ten minutes.

Take the temperature of the solution. Weigh [Quant.] into an evaporating-dish about 25 g. of the solution. Evaporate to dryness on the water bath and weigh again. Calculate the weight of dichromate dissolved by 1 liter of distilled water at the temperature observed.

*b.* Take 6 g. of the dichromate and boil with 10 c.c. of water in a test-tube. Is the solubility different? Allow the clear solution to cool (?). Explain the result.

*c.* Take 5 c.c. of water in each of two test-tubes. Shake one with crystallized, hydrated, sodium sulphate (previously powdered in a mortar) until no more dissolves. Simultaneously shake the other with powdered, anhydrous, sodium sulphate (freshly made by heating the hydrated salt in a small evaporating dish) until equilibrium is reached. In the intervals of shaking place both test-tubes in a beaker of water until their temperatures are equal. Ascertain the solubility of each kind of sodium sulphate as in *a*. Interpret the result [R].

*d.* Shake some calcium sulphate with recently boiled distilled water. Ascertain whether any of the salt has gone into solution or not. Repeat with chalk (calcium carbonate), rejecting the water with which it is first shaken (?).





*e.* Two immiscible solvents. Place one small particle of iodine in each of three test-tubes and add to one water, to the second potassium iodide solution, to the third ether, and shake each (?). If in any of the tubes iodine remains undissolved, pour off that solution into a clean test-tube. Now add a little ether to the first two, shake again (?), and describe carefully what seems to have happened. Can you deduce from this the relative solubility of iodine in the three solvents?



## CHAPTER VIII.

### CHLORINE AND HYDROGEN CHLORIDE.

#### 1. PREPARATION OF CHLORINE [Hood].

Experiments 1, *b* and 2 must be accomplished at one exercise. 1, *a* may be postponed to facilitate this.

*a.* Prepare some strips of filter paper by dipping them in starch emulsion to which one drop of potassium iodide solution has been added.

Place small quantities of potassium chlorate, minium, litharge, and potassium dichromate in as many test-tubes, and add a little concentrated hydrochloric acid to each. Hang a strip of the prepared paper in each tube (?) and notice also the odor (?). If no action takes place in the cold, apply heat. What property is common to the substances which cause the production of chlorine? Can you lay your finger on the cause of the difference in the behavior of the two oxides of lead?

*b.* Arrange a 250 c.c. generating flask (Fig. 10) and a bottle to wash the gas, like that in Fig. 12. Use glass tubes with the shortest possible rubber connections here and in 3, *d* (hydrogen chloride), as rubber tubing is destroyed by these gases. Test the apparatus to see that it is air-tight. Charge the flask by placing in it a large handful of coarsely powdered [Iron mortar] manganese dioxide and enough concentrated hydrochloric acid to cover the dioxide. Fill the washing bottle one-third (not more) full of water (use of this?). Notice what happens in the cold before applying heat. Use a shallow vessel filled with boiling water (why not a sand bath or naked flame?) to warm the flask. Fill three bottles and one test-tube with the gas by downward displacement, covering them during the process with a card perforated to admit the tube [See note below]. The experiments described in 2 should be begun as soon as one bottle is full. Under what conditions could you collect the gas over water [R]? Give some of the reasons for the slowness of the action, remembering that the manganese dioxide is insoluble in water.

When sufficient chlorine has been collected, filter a part of the residue in the flask into an evaporating-dish, evaporate to dryness [Hood], and redissolve in about 100 c.c. of boiling water in a beaker. If the residue was yellow, from the pres-





ence of ferric chloride, add sodium hydroxide solution drop by drop to the boiling liquid. The precipitate will at first be brown (?). Stop as soon as the fresh formations of precipitate are white (?). Boil vigorously for a few minutes, filter with the assistance of a water pump [See note], evaporate the filtrate to one-fourth of its former bulk, and set it aside to crystallize (?). Keep the product for use in 3, *a*. If pink crystals are not obtained, the manganese may have been precipitated along with the iron. In that case the precipitate, which should be kept as a matter of precaution, may be redissolved in hydrochloric acid and the precipitation repeated.



Fig. 13.

*Notes.*—Be careful not to allow any of the gas to escape into the room. When the gas is not being collected, let it bubble into caustic soda solution in a test-tube. Empty the flask and bottle finally into the sink in the hood.

Much time may be saved, and better results obtained, by using a pump in filtering liquids and in washing precipitates. The apparatus is shown in Fig. 13. Use a small cone made of perforated parchment paper to protect the point of the filter paper. The pump, the safety bottle for protecting the solution from admixture with water from the pump, and the connecting tubes may be obtained from the storeroom [Temp. order].

## 2. PROPERTIES OF CHLORINE [Hood].

*a.* In one bottle of the gas scatter a pinch of finely powdered antimony (?) [R].

*b.* Take a clean piece of sodium, cut from it with a sharp knife a very thin slice about half an inch square (fingers and knife used in handling it must be dry!), and introduce it into a bottle of chlorine (?). Cover the bottle with a glass plate and examine after the action is over. Dissolve the deposit in

2 c.c. of water, allow it to crystallize on a watch glass, and examine with a lens (?).

*c.* Connect a glass tube, terminating in a capillary opening, with the illuminating gas supply, and lower a small, burning gas jet into the third bottle (?). Blow the breath into the bottle after withdrawing the jet (?).

*d.* Fill a test-tube with hydrogen from a Kipp's apparatus. Bring this mouth to mouth with a tube of chlorine and mix the gases by repeated inversion. (Take care not to expose the mixture to direct sunlight.) Hold the mouth of each tube to the Bunsen flame (?).

### 3. PREPARATION OF HYDROGEN CHLORIDE.

*a.* [Hood.] Place small quantities of ammonium chloride, barium chloride, mercuric chloride, and manganous chloride (obtained in 1, *b*) in as many test-tubes, and add a few drops of concentrated sulphuric acid to each. Describe what happens in each case. Blow moist air across the mouth of the test-tube (?). Lower a glass rod dipped in ammonium hydroxide solution into each [See note below]. Try the effect of heating. Remember that the solubility (physical) of the substance in sulphuric acid will largely determine the speed of the action. In case of difficulty, therefore, powder the substance finely and shake with the acid for some minutes before heating and testing.

*Note.*—The use of ammonia is not a specific test for hydrogen chloride. This gas can be employed only for ascertaining the presence or absence of any one of several gases which are capable of uniting with ammonia.

*b.* To a pinch of finely powdered sodium chloride add a strong solution of phosphoric acid and heat if necessary (?). Test with ammonia as before. The above remark about solubility applies also to this case.

Why is hydrogen chloride displaced completely in *a* and *b* by these acids under these conditions [R]? In answering consider the results of *c* and *d*.

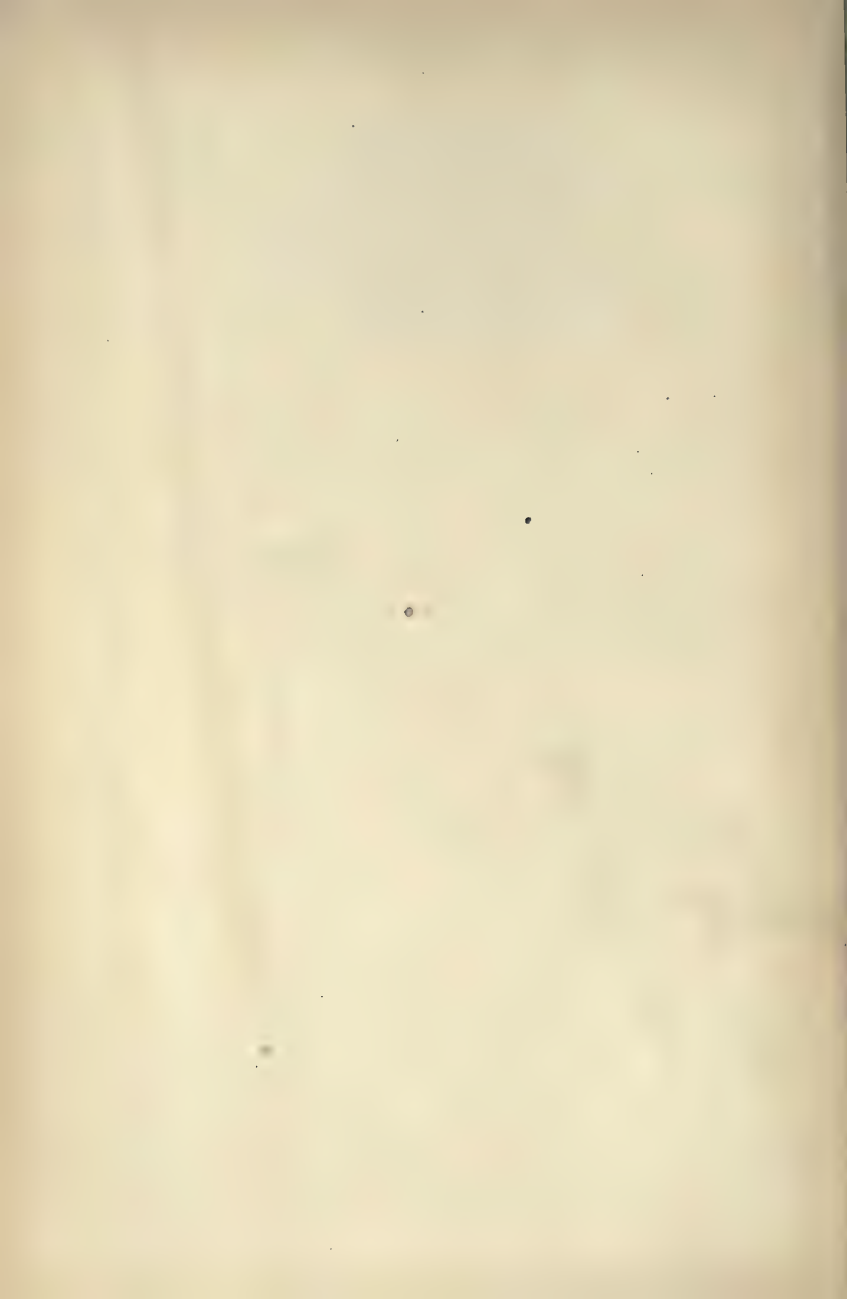
*c.* To a concentrated solution of sodium hydrogen sulphate add pure [Side-shelf] concentrated hydrochloric acid (?). Add the acid a very little at a time to avoid over-rapid precipitation, and agitate between additions. The longer the operation takes the better. Examine the result with a lens (?).

Relate this action to that in *d*, and consider both facts in answering the question in *b*.

*d.* [Hood.] In a 250 c.c. flask, fitted with dropping-funnel,







gas washing bottle containing sulphuric acid, and glass delivery tube, place a handful of common salt. Admit concentrated sulphuric acid through the funnel. Collect the gas in three dry bottles by downward displacement. Attach a nozzle to the tube and prepare an aqueous solution for use in 4, by passing the gas through 10 c.c. of water in a test-tube.

Relate this action to that in *c*.

#### 4. PROPERTIES OF HYDROGEN CHLORIDE AND HYDROCHLORIC ACID [Hood].

*a*. Invert one of the bottles of the gas in the pneumatic trough (?). Relate this property to that observed on blowing moist air into the gas (3, *a*). If any gas remains, what should you expect it to be? Test your conclusion experimentally.

*b*. Pour a little ammonium hydroxide solution on a slip of filter paper and plunge it into the second bottle (?).

Which of the kinds of chemical change does this illustrate?

*c*. Devise a way of proving, in a rough way, that the gas is heavier than air, and use the third bottleful for carrying it out.

*d*. Divide the aqueous solution into three parts. To the first add a piece of zinc (?). To the second add some sodium carbonate solution (?).

Why is carbonic acid displaced completely by the hydrochloric acid [R]?

Dilute the third part with an equal volume of water and distribute it between four test-tubes. To the first add blue litmus solution or paper (?); to the second a drop of silver nitrate solution (?); to the third a drop of mercurous nitrate solution (?); to the fourth a drop of lead nitrate solution (?). After the last three mixtures have settled, pour away the liquid, add water, and boil the precipitate with it (?). Afterward examine the liquids when they have become cold (?).

## CHAPTER IX.

### THE ATMOSPHERE, NITROGEN, AND AMMONIA.

#### 1. CONSTITUENTS OF AIR.

a. Place a large plug of copper turnings in the center of a hard glass tube about 25 cm. long. Fit with corks and short glass tubes, and connect with the short tube of the aspirator. Fill completely with water the bottle and long tube of the aspirator and close with a screw clamp. Arrange a vessel to catch the water discharged. Now heat the copper red-hot and then partly open the clamp so as to allow the water to be siphoned off in a slow stream. The air will pass over the heated copper. What change does the copper undergo and what collects in the bottle? After three-fourths of the water has run out, close the clamp, disconnect the hard glass tube, attach a delivery tube in its place, elevate the end of the siphon, and insert a small funnel. Pour water into the funnel and drive the gas over into a bottle of water inverted in the pneumatic trough. Ascertain whether this gas supports combustion. Describe the gas as regards color and smell.

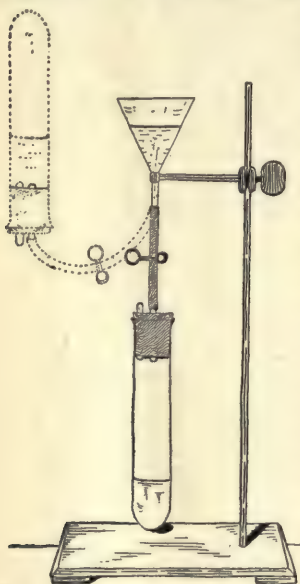


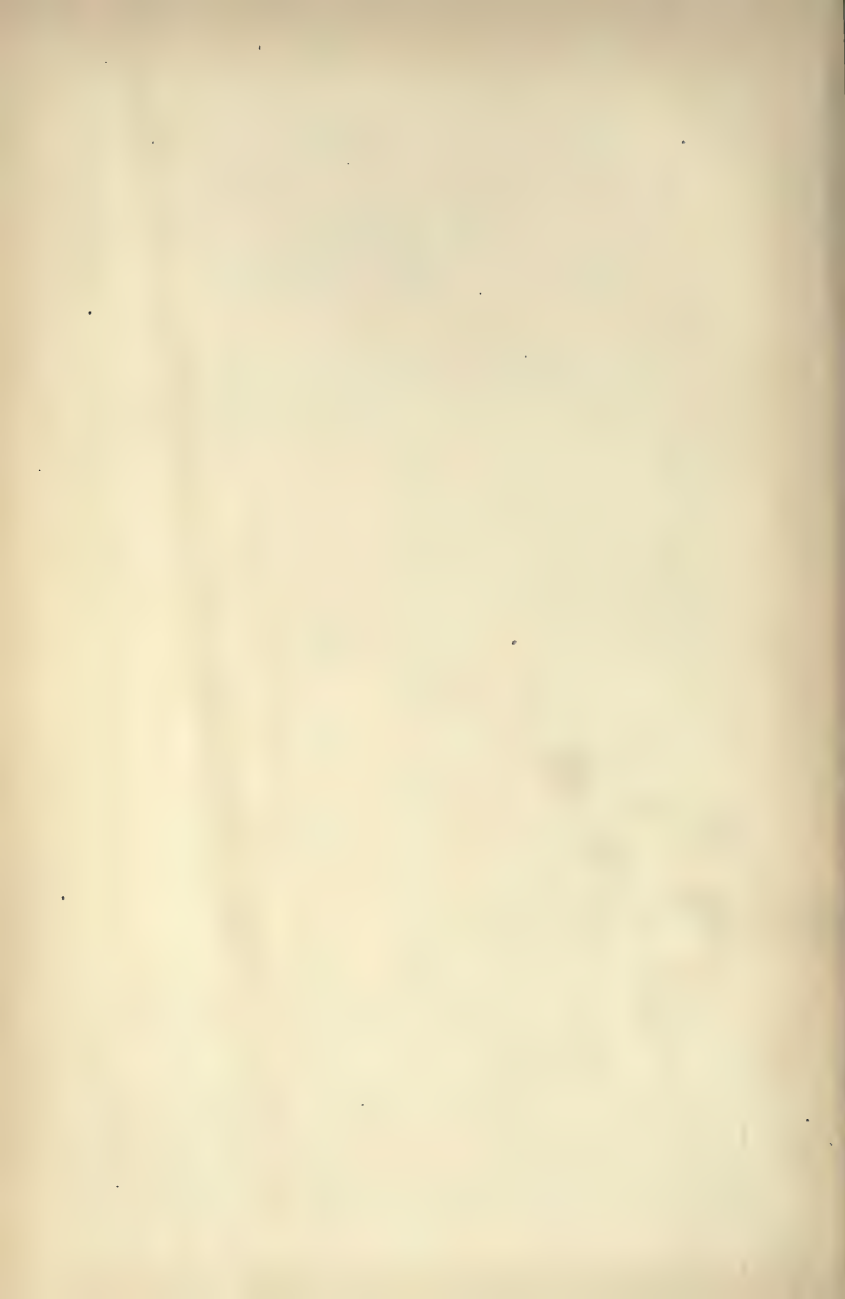
Fig. 14.

Other ways of removing oxygen from the air are described elsewhere (Chap. IV, 5, and in b).

b. PROPORTION OF OXYGEN TO NITROGEN BY VOLUME [Quant.]. (From Cooley's *Laboratory Studies*. Another method under nitric oxide, p. 66, 1, c.)

The apparatus (Fig. 14) consists of a large test-tube closed with a doubly bored rubber stopper, through which pass a piece of glass rod and a short glass tube terminating in a







nozzle. The latter projects but little beyond the stopper. The glass tube is connected with a funnel by means of a rubber tube 15 cm. long, which can be closed with a spring clamp. The stem of the funnel may be held in a clamp on the ring stand. Disconnect the test-tube, remove the piece of glass rod, pour the prepared alkaline solution of pyrogalllic acid<sup>1</sup> into the funnel, and open the spring clamp slightly so as to allow the solution to fill the entire rubber tube and nozzle. Push the rubber stopper tightly into the test-tube and then insert the glass rod and thus inclose a volume of air equal to the content of the test-tube and at the pressure of the atmosphere. During these operations, which should be performed as quickly as possible to avoid exhaustion of the solution by absorption of oxygen from the air of the room, do not warm the test-tube by handling. Now open the clamp and allow the liquid to enter. It will flow in until the oxygen is all absorbed. Keeping the clamp open, invert the test-tube and equalize the level of the liquid in both vessels; then close the clamp, restore the test-tube to its original position, and mark the levels of the liquid and the bottom of the stopper by means of rubber rings.

Disconnect the test-tube, wash out the liquid, and ascertain the volume marked off by each ring by weighing water. The smaller is that of the oxygen, the larger that of the air. Calculate the percentage composition of air by volume.

c. Place some clear barium hydroxide solution in the bottom of a beaker and leave it exposed to the air for some hours (?). What substances are found in the air besides those examined here [R]?

d. The weight of 1 liter of air, or of oxygen, or nitrogen, may be measured by the method described under carbon monoxide, p. 77, 9.

**2. NITROGEN.** Place about 8 g. of sodium nitrite and 3 g. of ammonium chloride in a 250 c.c. flask, add about 15 c.c. of water, and clamp the flask by the neck. Warm gently [Caution] and collect the gas in a bottle over water, after time has been given for the displacement of the air in the flask. Have an evaporating-dish filled with cold water in readiness and bring it up over the bottom of the flask when the action becomes too violent.

Ascertain whether this gas supports combustion, and describe it as regards color and smell. What impurity causes

<sup>1</sup>Prepared by dissolving 5 g. of pyrogalllic acid in 15 c.c. of water and adding a solution of 120 g. of potassium hydroxide in 88 c.c. of water.

the slight odor? With what substances will nitrogen unite directly [R]?

**3. AMMONIA.** Provide a small flask connected with a plain U tube. Place a little water in the bend of the latter so as to close the passage. Put into the flask a mixture of powdered quicklime and ammonium chloride, and heat (?). Preserve the solution of ammonium hydroxide. This reaction between strong bases and ammonium salts is used as a test for the latter.

What method would you pursue if you were asked to determine the weight of a liter of ammonia gas? How is the composition of ammonia by volume determined [Lect.]?

**4. AMMONIUM HYDROXIDE.**

*a.* Wash a piece of red litmus paper with water, dip it in the solution (?) and expose it to the air for some time (?). Dip a piece of blue litmus paper in dilute hydrochloric acid (?) and expose it to the air for the same length of time (?). Compare the results and interpret. Hold a rod dipped in concentrated hydrochloric acid over the solution (?).

*b.* Place a little of the solution in an evaporating-dish and leave it exposed to the air for twenty-four hours, noticing the odor from time to time (?). In a second evaporating-dish warm some of the solution and notice the odor from time to time [Hood] (?).

*c.* Neutralize the rest of the solution with dilute sulphuric acid and evaporate to dryness (?). Scrape out the residue and heat it strongly on the inverted lid of a crucible (?).

After completing Chap. XII, enumerate the things which *a*, *b*, and *c* show the solution of ammonia to contain. Explain fully the mechanism of the changes in *b* and *c*.

*d.* Place some ammonium chloride in the middle of an open, hard glass tube. Support this in a very slightly inclined ( $5^\circ$ ) position, put a moist piece of neutral litmus paper (or a piece of each color) in each end, and heat the salt strongly (?). Watch closely the effect on each test-paper (?). What is the action of heat on ammonium chloride? Which gas appeared first at the ends of the tube, and why first at both lower and higher ends? Has gravity any influence on the result?





## CHAPTER X.

### BROMINE, IODINE, AND FLUORINE, AND THEIR COMPOUNDS WITH HYDROGEN.

#### 1. PREPARATION OF BROMINE AND IODINE.

*Note.*—Chlorine and the elements studied in this chapter form a group having very similar properties, and are called the halogens. Recall the facts about chlorine and hydrogen chloride (Chap. VIII) and use them as a guide in trying to understand the chemistry of the rest of the group. Remember particularly that chlorine is colored, has a powerful odor, and does not cause fumes in moist air; and that hydrogen chloride is colorless and causes dense fumes in moist air. The corresponding substances throughout the group may be expected to present properties like these. The hydrogen compounds, hydrogen chloride, hydrogen bromide, etc., are known as the hydrogen halides.

*a.* For the action of manganese dioxide on hydrobromic and hydriodic acids (*cf.* Chap. VIII, 1, *b*, p. 30) see 5 and 6.

*b.* Powder about 1 g. each of the bromide and iodide of potassium, mix them in separate test-tubes with the same weight of powdered manganese dioxide, and moisten each with concentrated sulphuric acid. Warm very gently (?). How does a fluoride behave under these circumstances, and why [R]? Answer the same question in regard to chlorides (Chap. VIII, 3, *a*, and 1, *b*, pp. 32 and 30).

What have you ascertained about the solubilities of iodine (Chap. VII, 7, *e*, p. 29)? The relative solubilities of bromine are similar.

#### 2. THE HYDROGEN HALIDES: PRELIMINARY [Hood].

*a.* Powder about a gram of iodide of potassium, place it in a test-tube, and moisten it with concentrated sulphuric acid (?). Warm, if necessary. Investigate the result, which furnishes a mixture of gases, as follows:

*a.* Breathe across the mouth of the test-tube to ascertain the effect of the gas on moist air. What gas previously made showed the same behavior? Remembering the similarity between the halogens and between their corresponding compounds, what do you infer in this case? To confirm this conclusion, lower a glass rod dipped in ammonium hydroxide solution into the test-tube (?); also a strip of filter paper dipped in lead nitrate solution (?) [R].



$\beta$ . What is the color of the gas, or any part of it? What is the colored body? Was there any corresponding product when sulphuric acid acted on a chloride? By what kind of chemical action could this colored substance be formed from the one identified in  $\alpha$ ?

$\gamma$ . Study the odor of the gas and describe it (?). Was there any effect on the lead nitrate which remained unexplained in  $\alpha$ ? Can you now explain it [R]?

The work in  $\alpha$  and  $\beta$  leads to the recognition of two distinct gaseous products. That in  $\gamma$  will yield one, and perhaps two others. Still another distinct solid product may be observed on the walls of the tube (?). Construct separate equations representing the formation of the first product from the original materials, and of each of the others from this product and sulphuric acid. What two properties of sulphuric acid and what property of hydrogen iodide are illustrated by this set of experiments?

$b$ . Repeat the work in  $\alpha$ , using powdered potassium bromide instead of the iodide, and answer the same questions, substituting the words bromine and bromide everywhere for iodine and iodide.

$c$ . Cover a square of glass with a thin layer of paraffin by warming it very cautiously far above a Bunsen flame and rubbing it on one side with solid paraffin. Moisten about 3 g. of flourspar in a leaden dish [Temp. order] with concentrated sulphuric acid (do not cover with the acid). Remove part of the paraffin from the glass by drawing some design with the end of a file, thus exposing part of the glass to the action of the vapor. Place the glass on the top of the leaden dish and set the whole above a radiator, at such a distance that the dish will be warmed without risk of the paraffin being melted. After half an hour warm the glass once more and wipe the paraffin off with a piece of filter paper (?). Write equations representing the action, and state what becomes of each of the constituents of the glass [R]. Try the test of a rod dipped in ammonium hydroxide solution and held over the contents of the lead dish (?). Is there any odor or action on paper dipped in lead nitrate solution, indicating reduction of the sulphuric acid, similar to that caused by hydrogen iodide or bromide?

How does a chloride behave under similar circumstances? What difference between the four hydrogen halides do the results in  $\alpha$ ,  $b$ , and  $c$  bring to light? Arrange the hydrogen halides in the order of their stability toward oxidizing agents, so far as your experiments enable you to judge of this.







*d.* Take small samples of finely powdered bromide and iodide of potassium in separate test-tubes, cover each with concentrated phosphoric acid solution, and agitate for some time (?). Note the apparent slowness of the action on account of the insolubility (physical) of the salts in this liquid (the sodium salts might be substituted with advantage, as they are much more soluble). Warm, if necessary. Observe the odor and apply the ammonia (?) and lead nitrate tests (?) as above. What difference between the actions of sulphuric and phosphoric acids do you observe? What do you infer in regard to the properties of these acids?

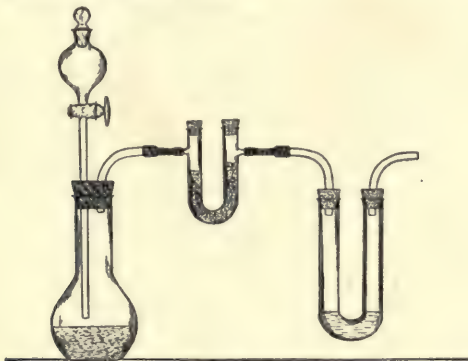


Fig. 15.

### 3. PREPARATION OF HYDROGEN BROMIDE AND IODIDE [Hood].

*a.* Fit up a 250 c.c. flask with a dropping-funnel and tube, and connect with a U tube (Fig. 15). Fill the latter with dry, broken glass, or porcelain, mixed with a little red phosphorus (why?). Connect the other limb of the U tube with a second, larger U tube [Temp. order] containing about 10 c.c. of water. Place about 5 g. of red phosphorus mixed with twice its weight of sand in the flask, add 5 c.c. of water, and mix by shaking. Pour into the globe of the funnel about 8 c.c. of bromine. Allow the bromine to flow drop by drop on to the phosphorus, and let the gas dissolve in the water in the second U tube. Record such properties of the gas as you observe. Keep the solution for use in 4, *a* and 6.

*b.* Hydrogen iodide may be made in the same way, excepting that the iodine and phosphorus (in the proportion of 11:1 by weight) are first intimately mixed in a mortar and are then placed in the flask, and the water is allowed slowly to drop upon them from the funnel.

**4. DISPLACEMENT OF HALOGENS BY EACH OTHER** [Hood]. Prepare a little chlorine (p. 30, 1, *b*) in a side-neck test-tube, fitting the latter with a tube bent at right angles.

*a.* Pass a few bubbles of chlorine into a diluted (1:10) solution of potassium bromide in a test-tube (?). Add a drop or two of the liquid to a test-tube full of starch emulsion (?). Add a little chloroform or ether to the remainder of the liquid and shake (?). Explain (see notes). Pass a few bubbles of chlorine through a small portion of the solution of hydrogen bromide obtained in 3, *a* (?).

*b.* In like manner pass a few bubbles of chlorine through a very highly diluted solution of potassium iodide (?). Add a few drops of the liquid to a test-tube full of starch emulsion (?). Add a little chloroform or ether to the remainder of the liquid and shake (?). Explain. In case the aqueous solution remains brown (why? R) use a little more chlorine and shake again (?).

*c.* Examine the action of bromine water on highly diluted potassium iodide solution in exactly the same way (?).

*Notes.*—When layers of different colors appear in a test-tube, always mix the materials by inverting the tube once or twice before drawing conclusions. The contents of the whole test-tube should be made as homogeneous as possible.

Chloroform and ether have no chemical action on bromine or iodine; they simply dissolve them more readily than water does.

**5. DISPLACEMENT OF SULPHUR AND OXYGEN BY THE HALOGENS AND BY EACH OTHER.**

*a.* Place 2-3 c.c. of diluted sodium sulphide solution in a test-tube and add bromine water (?).

*b.* [Hood.] Place 5 g. of powdered iodine with 50 c.c. of water in a small flask provided with a cork and a tube, bent at right angles and extending to the bottom. Pass hydrogen sulphide from a Kipp's generator through the mixture, loosening the cork once or twice at first to permit the air to be displaced by the gas, until the iodine is all gone and the solution no longer becomes brown on being shaken. Agitate constantly to hasten the process. Describe what happens. Warm and filter the solution.

Obtain a distilling flask and condenser [Temp. order] and distil the filtrate fractionally (Fig. 16), collecting first the part that comes over at 100°, then the parts boiling between 100-103°, 103-106°, and so forth. Use a very small flame and be careful not to allow it to reach the walls of the flask above the liquid or breakage will take place. A large flame may not only crack the flask, but may also cause the thermometer to







show a higher temperature than it could acquire from the vapor alone. Stop when the liquid is nearly all distilled off. Note the highest temperature reached. Pour the residue into a test-tube and keep the series for use in 7.

What substance causes the color of the higher fractions and of the residue?

Recall the action of chlorine on water in sunlight. What is the action of fluorine on water [R]? In what way do these

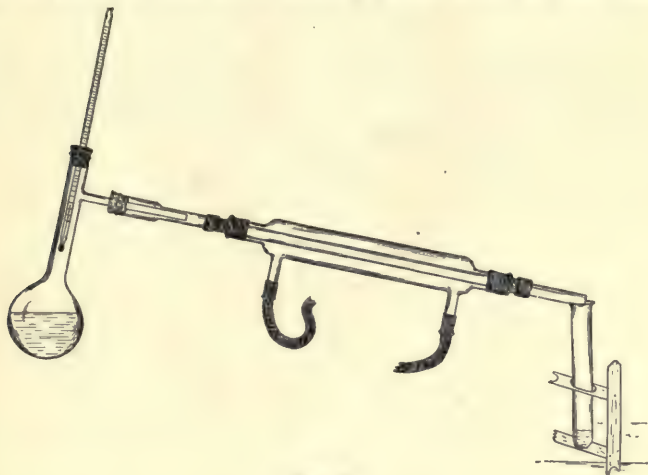


Fig. 16.

resemble the action of iodine on hydrogen sulphide? What is the action of oxygen on hydrogen sulphide solution [R]? What is its action on hydrogen iodide solution [R]?

**6. PROPERTIES OF AQUEOUS HYDROBROMIC ACID.** Divide the solution into six portions and examine its behavior toward (a) litmus, (b) zinc in contact with a platinum wire, (c) silver nitrate solution, (d) mercurous nitrate solution, (e) lead nitrate solution, (f) powdered manganese dioxide (warm). Boil c, d and e, after pouring away the supernatant liquid and adding more water to each (?). Compare these results with those found in the case of hydrochloric acid, (p. 33, 4, d).

The solution is as good a conductor of electricity as that of hydrogen chloride. What conclusion do we draw from this [R., p. 47 1, e]?

**7. PROPERTIES OF AQUEOUS HYDRIODIC ACID.** Add silver nitrate solution to each of the fractions obtained in 5, using

only a part of the liquid in the case of the two with the highest boiling points. At what temperature did the most concentrated solution of hydrogen iodide come over? What peculiarity of aqueous hydriodic acid does the result indicate? What other solutions show the same peculiarity? Compare with the behavior of ammonia (p. 36, 4, *b*).

Place a piece of zinc, in contact with a platinum wire, in the remainder of one of the higher fractions (?). Test the other with litmus paper (?), and then add powdered manganese dioxide and warm (?).

**8. IDENTIFICATION OF HALOGEN COMPOUNDS.** On the basis of the experiments in this chapter, devise a system of tests which would enable you to distinguish between chlorides, bromides, iodides, and fluorides.





## CHAPTER XI.

### OXYGEN COMPOUNDS OF THE HALOGENS. OZONE AND HYDROGEN PEROXIDE.

**1. HYPOCHLOROUS ACID AND HYPOCHLORITES.** Fit up a chlorine apparatus capable of delivering a large amount of chlorine. Use a gas washing bottle (why?) containing a small (why?) amount of water. Use the same source of chlorine in 1, 3, and 5, *a*.

*a.* Make an aqueous solution of chlorine in a test-tube. Retain a few drops of this for use in *b* and place in the remainder some litmus paper, paper with printing and pen and pencil marks upon it, and a piece of colored calico. Observe the effect on each. Explain [R].

*b.* To a few drops of chlorine water add a drop of indigo solution (?).

*c.* Slake a piece of quicklime in a small beaker and add enough water to make a thin paste. Pass chlorine into the mixture for ten or fifteen minutes (?), cooling the vessel by surrounding it with water (why?) and stirring the contents during the process. Thoroughly soak a piece of litmus paper (?) and a piece of colored calico (?) in the paste and then place them in dilute sulphuric acid (?). Repeat this treatment of the litmus and calico if necessary.

How could you make a solution of pure calcium or potassium hypochlorite [R]?

**2. NASCENT ACTION.** Dilute some potassium permanganate solution with water, and add an equal volume of dilute sulphuric acid to it. Divide into two parts. Through one pass a stream of hydrogen gas from a Kipp's apparatus (?). To the second add some zinc dust (?). Interpret the result.

**3. CHLORATES** [Hood]. Dissolve about 3 g. of solid potassium hydroxide in 7 c.c. of water in a test-tube and saturate (test? The solution must cease to feel soapy) the hot solution with chlorine. Crystals will appear during the process and will increase in quantity when the liquid cools. Describe the crystals. Filter, acidify (test?) the filtrate with pure nitric acid, and test with silver nitrate solution (?). Dry the crystals, heat them in a narrow tube, and test for oxygen (?).

To potassium chlorate solution add silver nitrate solution (?). To a minute amount of potassium chlorate add a few drops of pure, concentrated hydrochloric acid [R] (?).

**4. PERCHLORATES.** Measure 500 c.c. of water into your 1-liter bottle, and mark the level reached. Observe the temperature and pressure of the air and calculate the weight of potassium chlorate which will be necessary to give this volume of oxygen while leaving the perchlorate and chloride behind. This stage is reached when one-fifth of the total oxygen has been evolved. Weigh this quantity of chlorate into a rather wide hard glass test-tube fitted with a cork and delivery tube. See that the apparatus is air-tight. Heat the chlorate and drive off enough gas to fill the bottle to the mark by displacement. Proceed slowly toward the end so as to allow the gas to cool, stop heating when the mark is reached, and remove the tube at once from the water. Pour the melted substance into a mortar before it has time to solidify. Powder the mixture.

To separate the substances, calculate approximately the amount of potassium chloride which must be present, and shake the powder persistently with an amount of cold water just sufficient to dissolve it (see note). Collect the undissolved material on a filter paper just large enough to hold it, and wash it with a few drops of cold water. Calculate the amount of water which at 100° will dissolve the residue, assuming it to be potassium perchlorate. Dissolve it in this amount of water by boiling, and allow the solution to stand for an hour or two. Collect the crystals, wash them as before, and dry them on a radiator.

Dissolve a little of the substance in distilled water and test with silver nitrate solution (?).

To a minute amount of the crystals add a few drops of pure, concentrated hydrochloric acid (?). How could you distinguish between a perchlorate and a chlorate?

*Note.*—The solubilities of the salts are as follows:

	Grams dissolved by 10 c.c. of water.		
	15°	20°	100°
Potassium chloride	3.3	3.5	5.6
Potassium perchlorate	0.15	0.18	2.0

#### 5. BROMIC AND IODIC ACIDS [Hood].

*a.* Take two test-tubes and place in each 5 c.c. of water. Add a single drop of potassium iodide solution to one, and a single drop of potassium bromide solution to the other. Pass chlorine, a few bubbles at a time, into them alternately, shaking after each addition of chlorine (?). Continue until no further change occurs. Explain the changes which are observed [R].







*b.* Take 2 c.c. of potassium bromate solution in a test-tube, add an equal volume of pure, dilute sulphuric acid (?), and divide into two parts. Allow one part to stand to see whether there is any further action (?). To the other part add a single small fragment of iodine and shake for several minutes (?). Pour the solution away from any undissolved iodine and shake the former with a few drops of chloroform (?). (The chloroform is used simply as a solvent which collects the free halogen from dilute solution in a great bulk of water). Which of these halogens has the greater tendency to unite with oxygen?

**6. OZONE.** Place two or three pieces of phosphorus in a bottle, add enough water to half cover them, and one drop of potassium dichromate solution. Dip a strip of filter paper in starch emulsion to which a drop of potassium iodide solution has been added. Fasten it by means of a cork in such a way that it hangs down almost to the phosphorus. Allow the apparatus to stand for an hour (?). After the effect appears on the starch paper, try a paper dipped in manganous sulphate solution (?). Return the phosphorus to the supply bottle.

#### 7. PEROXIDES.

*a.* Dissolve about 1 g. of sodium peroxide (how is this made?) in 100 c.c. of cold water in a flask. Add this amount of the oxide a very little at a time, shaking and cooling (why?) the mixture in a stream of water during the process. What does the solution contain [R]? Add to it a little dilute sulphuric acid a few drops at a time, continuing the cooling. What does the solution now contain? Divide the mixture into three parts.

*b.* To the first, placed in a narrow test-tube, add dilute, acidified [*cf.* p. 57, 3, *c*] potassium permanganate solution (?). Test the gas which comes off for oxygen, if the quantity is sufficient for the purpose. By what term should we describe this effect of hydrogen peroxide?

Add the second portion to starch emulsion containing a drop of potassium iodide solution (?). By what term should we describe this effect of hydrogen peroxide?

To the third add some ether (object of this [R]?) and shake, and then add one drop and no more of potassium dichromate solution and shake again (?).

*c.* Suspend lead dioxide, barium dioxide, and manganese dioxide separately in water and treat with dilute sulphuric acid as in *a*. Filter and apply to each filtrate the ether and dichromate test as in *b* (?). What are the differences in behavior and constitution between a true peroxide and those oxides which are sometimes called peroxides [R]?

## CHAPTER XII.

### IONIC CHEMICAL ACTIONS. INTERACTIONS OF ACIDS, BASES, AND SALTS.<sup>1</sup>

1. IONIZATION. How do we ascertain experimentally whether a substance is ionized in solution or not and learn the extent of the ionization [R]? The degrees to which aqueous solutions of many substances are ionized are given in a table in the Appendix. Constant reference to this will be necessary in interpreting the observations in this and succeeding chapters.

The experiments of paragraph 1 are really selected from several later ones and are performed as a group, since the same apparatus serves for all. They may be postponed if a set of the apparatus is not available at the moment.

Obtain [Temp. order] a pair of electrolytic cells like that in Fig. 17, and half fill one with dilute sulphuric acid. When the cells are connected in series with the battery, evolution of gas in this cell will indicate that the material in the second cell is a conductor, and that the circuit is therefore complete. In the contrary case, the second substance is a non-conductor, or at all events a very bad conductor. If the substance in the second cell is a solution, what conclusion may be drawn in regard to the condition of the dissolved body in each event [R]?

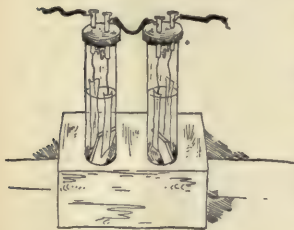


Fig. 17.

Half fill the second cell with the substances named below in turn. See very particularly that the electrodes in each cell are on opposite sides of the glass partition, connect with the battery, and observe the effect in the first cell. When the same experiment has been shown in the lecture-room, the result may be recorded here and the experiment omitted. Wash the cell and electrodes very carefully after each trial.

<sup>1</sup> Class-room instruction, illustrated experimentally, must precede and accompany the work in this chapter or little will be learned from it. The sign for reference to a book or to information obtained in lectures [R], has been used very little in this chapter, as the dependence on these sources is constant.







The substances, or solutions, in *a*, *c*, *d*, *e*, *f*, show the behavior typical of the classes of substances, or their solutions, to which each example belongs. After giving the result in your notes, name the class of bodies which is illustrated in each case.

- a*. Dry crystallized sodium chloride.
- b*. Distilled water.
- c*. Aqueous solution of sodium chloride.
- d*. Diluted aqueous solution of sodium hydroxide.
- e*. Diluted aqueous solution of hydrochloric acid.
- f*. Aqueous solution of sugar.

*g*. Dry the cell by washing first with alcohol and then with ether [Instructions]. Test the conductivity of a solution of dry hydrogen chloride in toluene [Side-shelf]. What difference between water and toluene do *e* and *g* bring to light? Keep the solution in a dry test-tube for further use in *2, d*.

*h*. Subsequent chemical experiments with the same, or similar, substances or solutions will bring out the relation between conductivity and chemical behavior and its explanation.

The chemical composition of the ions into which any compound is split is ascertained by examining the substances set free at the electrodes during electrolysis of a solution and by a study of the interactions of the ions in the solution. The use of the latter method is illustrated in later paragraphs.

Solutions which show exceptional depression of the freezing point, are decomposed by a current of electricity, and exhibit certain other properties capable of quantitative correlation, are called *electrolytes*. Substances which, when dissolved in water, furnish electrolytes are called *ionogens*. Acids, bases, and salts, and no other compounds, are ionogens. Solutions of ionogens in water are *mixtures* of several physical components. These components are related in sets to one another through *chemical equilibria*. For example, in water

alone there are three components ( $\text{H}_2\text{O} \rightleftharpoons \overset{+}{\text{H}} + \overset{-}{\text{OH}}$ ), in hydrochloric acid there are five components in two sets ( $\text{H}_2\text{O} \rightleftharpoons \overset{+}{\text{H}} + \overset{-}{\text{OH}}$  and  $\text{HCl} \rightleftharpoons \overset{+}{\text{H}} + \overset{-}{\text{Cl}}$ ). With certain restrictions each component of such a solution may *undergo chemical change independently* of the others.

Give a complete list of the different things (eight of them) which are present in every aqueous solution of common salt, distinguishing those which are present in large from those which are present in minute quantity. How do the propor-

tions of each of these things vary with changes in the concentration of the solution? Which of the eight are usually most conspicuous in chemical actions involving solutions of common salt [R]? State what happens when these most active constituents are removed from the scene of action [R. Lect.]. What you have stated about common salt holds *mutatis mutandis* for solutions of all other ionogens.

## 2. BASES AND ACIDS: PROPERTIES OF HYDROXIDION AND HYDRION.

a. Examine distilled water in respect to (a) taste, (b) behavior with litmus, (c) conductivity (*cf.* 1, b).

b. Dissolve a small piece of sodium hydroxide in water and examine the solution in respect to (a) taste, by diluting a little and tasting one drop, (b) behavior with litmus, (c) conductivity (*cf.* 1, d). These properties belong to aqueous solutions of all bases. What component alone is common to all such solutions and has the above properties?

How can sodium hydroxide be obtained in solid form, starting from sodium and from sodium oxide respectively [R]? Leave a small piece of the solid exposed to the air for 24 hours. Examine the result (?) and add excess of hydrochloric acid (?). Explain.

c. Examine an aqueous solution of hydrochloric acid in respect to (a) taste, (b) behavior toward litmus, (c) conductivity (*cf.* 1, e), (d) action on a piece of marble, (e) action on an iron nail (clean this with sandpaper before use). These properties are shown by all aqueous solutions of acids. What component alone is common to all such solutions and has these properties?

How is the solution of this acid made?

d. Take the solution of hydrogen chloride in toluene and examine it in respect to (a) conductivity (*cf.* 1, g), (b) action on a piece of marble, dried in advance by heating in a dry porcelain dish for a few moments, (c) action on an iron nail (clean as before). Be sure that perfectly dry vessels are used in these experiments. Compare and interpret the results in c and d. What body whose presence might have been expected is absent from this solution?

e. Recall the action of concentrated sulphuric acid on zinc (p. 22, 1, e) and the action of dilute sulphuric acid on zinc (p. 22, 1, c). Recollect that the former is a poor conductor and the latter a good conductor. To what inference does the last fact in itself lead? Can you explain the two different chemical actions by the help of this inference? What body





is acted upon by the zinc in the former action? To what class of actions does this belong? What are the five components of dilute sulphuric acid? Which of them is affected by the zinc and how?

### 3. SALTS: PHYSICAL PROPERTIES OF THEIR IONS.

*a.* Examine a solution of potassium bromide. What is the color of bromidion? Take a minute amount (say 0.2 g.) of cupric bromide in a dry test-tube. Add two drops of water and agitate for some time (?). Then add more water, a drop or two at a time, agitating vigorously and giving the substance time to dissolve, if it can, after each addition. Continue the addition of water cautiously until the substance has all dissolved and afterward until the change in color is complete, and then stop. What is the color of the molecules of cupric bromide? What is the color of cuprion? Compare the color with that of cupric sulphate solution (?). Now add solid potassium bromide to the solution and shake vigorously (?). Interpret the result.

*b.* Make solutions of chrom-alum, cobalt chloride, potassium permanganate, and potassium dichromate from the solids and dilute to see whether any change in color occurs. What are the colors of trichromion, cobaltion, permangananion, and dichromanion? These colors are useful for purposes of recognition.

*c.* Aqueous solutions of substances of this kind all conduct electricity (*cf.* 1, *c*).

**4. IONIC CHEMICAL CHANGES: I. NEUTRALIZATION.** When dilute solutions of two ionogens are mixed, no appreciable change occurs, unless one (or both) of the two new ionogens which may be formed by union of the ions crosswise is a little ionized substance. In this latter event however a noteworthy chemical change is encountered. Such chemical changes, producing soluble products, are illustrated in 4, 5, and 6.

Dissolve about 10 g. of sodium hydroxide in 100 c.c. of distilled water. Place the solution in a burette. Take about 6 c.c. of concentrated hydrochloric acid in an evaporating-dish, add about half its volume of distilled water, and then two drops of phenolphthaleïn solution, and allow the alkali to run in drop by drop until the last drop confers the faintest perceptible pink tinge on the whole solution. Repeat the experiment, if you do not at first succeed in stopping at the right point. Concentrate the solution on the sand bath until a drop deposits crystals on cooling, and then remove the dish from the sand bath promptly and set it aside. When sufficient



crystals have appeared, dry them with filter paper and examine with respect to (*a*) form, (*b*) taste, (*c*) exposure to moist air, (*d*) action of a solution on litmus, (*e*) conductivity of aqueous solution (done already, 1, *c*). Construct a table comparing the substance in these respects with the materials from which it was made. Compare the substance with common salt on the side-shelf. How would you determine whether a substance obtained in this way contained "water of crystallization" or not? Make the necessary experiments (?). Wash out the burette.

Name all the physical components of the solutions of the acid and base used in the present experiment. In what relative proportions are they present (see Appendix)? When the two systems are mixed, which components will unite to form new bodies, and to what extent (see Appendix)? Which of these changes will be the most extensive? Express this change in symbols. What is the cause of the completeness of the action?

The first experiment on the law of definite proportions (p. 10, 2, *a*) presents another case of neutralization. Answer the same questions in regard to this action.

Define neutralization in terms of the ionic theory.

**5. PARTIAL NEUTRALIZATION: ACID SALTS.** Fill one burette with potassium hydroxide solution. Add 30 c.c. of concentrated sulphuric acid to 70 c.c. of water in a beaker and fill the other burette with the cold, diluted acid. Ascertain, as in 4(*q. v.*), what volume of the alkali will neutralize 5 c.c. of the acid, concentrate by evaporating to about 12 c.c., remove the dish from the steam bath, and allow the resulting solution to crystallize. Dry the crystals on filter paper. To a second portion of the acid, twice as great as before (10 c.c.), add exactly the same amount of alkali, evaporate to about 5 c.c., and treat as before.

Compare the two lots of crystals as regards (*a*) form, (*b*) taste, (*c*) reaction of solution with litmus, (*d*) "water of crystallization." Confirm by examining specimens of the same substances from the side-shelf. Wash the burettes very carefully to prevent the stopcocks becoming fast.

Name all the things (five of them) which the mixture known as a solution of potassium hydroxide contains: also all the things (five of them) which the mixture known as dilute sulphuric acid contains. Describe what happens to each of these, so far as they are changed in the course of the titration, expressing the changes in equations. If the solution had been one of ammonium hydroxide, what additional kind of material would have been present and what changes would it







have undergone in the course of the neutralization? Express these changes also in equations.

[Advanced students.] Make a solution of sodium bicarbonate and test it with litmus (?). Why do some acid salts show no acid reaction with litmus? Define the terms "normal salt" and "acid salt." Test cupric sulphate and ferric chloride solutions with litmus (?). Why are some normal salts basic and others acid in their reactions toward litmus? Answer these questions in terms of the ionic theory.

What is meant by the term "basic salt?" Give examples [R].

*Note.*—Aqueous solutions of suitable concentration of the substances used in all experiments will be found on the side-shelf. These should be used, excepting where the making of a solution is expressly directed. Examine the same substance in the solid form when you fetch the solution.

## 6. IONIC CHEMICAL CHANGES: II. FORMATION OF "WEAK" BASES AND ACIDS.

*a.* Formation of an hydroxide. To some ammonium chloride solution in a test-tube add some sodium hydroxide solution (?). Observe the odor.

Name all the physical components of each solution and the relative proportions in which they are present (see Appendix). When the two solutions are mixed, which components will unite and form new bodies, and to what extent (see Appendix)? Which of these changes will be the most extensive? Express this change in symbols.

Formulate also the process by which, here, ammonia is set free. Define the term "weak base" and illustrate.

This kind of action is often accompanied by precipitation, as in making zinc hydroxide. This is formulated as follows:



On what two things does the completeness of such an action depend? Which exercises here the decisive influence [R]?

*b.* Formation of an acid. Add a few drops of concentrated sulphuric acid to a concentrated solution of sodium acetate in a test-tube and, if necessary, warm gently (?). Observe the odor.

Answer the same questions as in the second paragraph of *a.*

Define the term "weak acid" and illustrate.

Give a list of acids which might be formed by union of the proper ions after the manner of acetic acid (see Appendix).

This kind of action is often accompanied by precipitation as in making silicic and boric acids [R]. Formulate the action producing metasilicic acid from sodium metasilicate on the model given above. Which is here probably the chief cause of the completeness of the action?

**7. IONIC CHEMICAL CHANGES: III. FORMATION OF SALTS.** With a few exceptions (illustrations? See Appendix), salts are all extensively ionized in dilute solutions. Decided chemical changes therefore are not produced by mixing their ions in solution. When, however, the molecules are sparingly soluble in water, precipitation occurs and almost complete chemical change may, nevertheless, take place. The following experiments deal with changes which occur on this account:

*a.* Take some of the ordinary calcium chloride solution in one test-tube. Dilute one drop of it with a large amount of water in another. Add a drop or two of dilute sulphuric acid to each (?). Explain.

*b.* Formation of salts. Place 3 or 4 c.c. of silver nitrate solution in a test-tube and dilute with water. Add potassium chloride solution cautiously, and agitate continuously, until no further precipitation occurs. Filter, concentrate the filtrate and pour it into a watch glass to crystallize. Two salts are formed. Make lists of the physical components of each solution. Write the general equation and also the two equations representing the formation of the salts separately from their components, and state when the formation of each mainly occurs. Is the formation of a precipitate theoretically essential in order that any change may take place between salts?

*c.* Take 3 or 4 c.c. of silver nitrate solution, dilute with water, and use one-fourth for each of the following experiments.

Add the first and second portions of the silver nitrate solution to solutions of calcium chloride and cobalt chloride respectively (?). What do these results show the solutions of these chlorides and potassium chloride to possess in common?

Add the third portion of the silver nitrate solution to a few drops of potassium chlorate solution (?). What conclusion may we draw from this result in regard to the ions present in the solution of potassium chlorate?

Chloroform and chloroacetic acid fail to give a precipitate with silver nitrate. How do these substances differ from chlorides?

Dilute a few drops of silver sulphate solution with water and add some potassium chloride solution (?). What does this show to be common to the solutions of silver nitrate and sulphate?







To the fourth portion of the silver nitrate solution add potassium cyanide solution [Care ! Poison !] until the precipitate at first formed is re-dissolved (see note). Now add a drop or two of potassium chloride solution (?). What is absent from this silver solution and present in those of silver nitrate and sulphate as judged by the potassium chloride test?

*Note.* — The solubility of an “insoluble” substance is not usually affected by the mere physical presence of another soluble substance, whether this be a new reagent or an excess of an old one. The only body playing the part of a solvent in such cases is water. When solution occurs the acid or other chemical reagent simply transforms by chemical change a precipitated material into a new substance soluble in water and has itself no share in promoting the physical process of solution. Here the new body is potassium argenticyanide,  $KAg(CN)_2$ . What do the ions of this substance appear to be?

*d.* Place in three test-tubes small quantities of solutions of sodium sulphide, sodium sulphate, and sodium thiosulphate, dilute each with water, and add to each a few drops of cadmium nitrate solution (?). Assuming that we know the nature of the precipitate in the first case and the ions in that case, what conclusion do we draw from the behavior of the other two solutions?

*e.* In what way does the lecture experiment with potassium ethyl sulphate and barium chloride illustrate the same points?

*f.* Put some highly diluted potassium permanganate solution in one test-tube and some diluted manganous chloride solution in another. Add to each a few drops of sodium carbonate solution (?). Why does the second behave differently from the first?

*g.* Place approximately equal small quantities of sodium hydrogen carbonate (bicarbonate) and potassium hydrogen tartrate in a mortar and rub them together (?). Now throw this mixture into a small beaker of water? The gas is carbon dioxide and comes from the spontaneous decomposition of the molecules of one of the products of the interaction. Test the solution for hydrion (?, test?).

To explain this action we must know the ions of the ingredients. Kalion and natrion may safely be assumed. Make separate solutions of the ingredients and test each for hydrion. Make lists of the physical components of each solution so far as these data enable you to do so. Which of these components will unite to form a new body (see Appendix)?

Formulate the action by which this body is formed and complete the explanation.

## 8. IONIC CHEMICAL CHANGES: IV. DISPLACEMENT OF ONE ION.

*a.* Place several pieces of granulated zinc in a dilute solution of cupric sulphate and set aside till the change is complete (?). Stirring will hasten the change (why?). Filter and examine the precipitate and filtrate (?) in turn as follows:

Treat the precipitate with a drop or two of concentrated nitric acid in a test-tube (?), and add ammonium hydroxide to the result (?). Treat a fragment of copper turnings in the same way (?).

Add colorless ammonium sulphide solution to the filtrate (?). What does it contain [R]?

*b.* In what way do the experiments in Chap. VI, 1 (p. 22) illustrate this kind of action? Answer the same question in regard to Chap. VI, 2, *a*, and *d* (p. 23).

How does the action in Chap. VI, 1, *e* differ from these and the present experiment? Which physical components were active in 1, *e*?

*c.* The order in which the metals stand in reference to their tendency to enter the ionic condition from the metallic (the order of decreasing "solution tension," known also as the "electro-motive series") is as follows:

Alkali metals [R].	Iron	(Antimony).
Alkaline earth metals [R].	Cobalt.	(Bismuth).
Magnesium.	Nickel.	Mercury.
Aluminium.	Tin.	Silver.
Manganese.	Lead.	Palladium.
Zinc.	<b>Hydrogen.</b>	Platinum.
Chromium.	(Arsenic).	Gold.
Cadmium.	Copper.	

Each of these metals will in general displace from a normal solution the ions of any metal below it in the series [R. Electro-Chemistry]. The places of the metals in parentheses are only approximate.

*d.* Examine your notes on Chap. X, 4 and 5 (pp. 40-41).

What is the action of free chlorine on bromide ions (4, *a*)?

What is the action of free chlorine on iodine ions (4, *b*)?

What is the action of free bromine on iodine ions (4, *c*)?

What is the action of free bromine on sulphur ions (5, *a*)?

What is the action of free iodine on sulphur ions (5, *b*)?

Arrange these four elements in a series similar to that for metals given above. Where would you place fluorine in this series? Can you indicate the approximate position of oxygen (5, *b*, last par.)?





**9.** What three distinct kinds of chemical change involving ions are illustrated in this chapter (*cf.* 1, 4, 5, 6, 7, 8)? Give examples of each. A fourth kind occurred in Chap. XI, 3 (p. 43), and again in 5, *b* (p. 45), and still again in 7, *b* (p. 45). What was this kind? The fifth kind will be met with in Chap. XIII, 3, *e* (p. 58), and in Chap. XV, 4, *c* (p. 69).

**10.** In previous lecture and laboratory experiments we have observed the formation of acids, bases, and salts in other ways than those illustrated in this chapter. These ways are non-ionic, or not distinctly ionic. Give illustrations of such of these ways as you recall: acids, two ways; bases, two ways; salts, three ways. Two other ways of making salts will occur later.

## CHAPTER XIII.

### SULPHUR AND ITS COMPOUNDS.

#### 1. SULPHUR.

*a.* Place a few grams of sulphur in a dry test-tube and heat slowly with a small flame until the substance boils. Describe all the changes which occur.

*b.* Pour the boiling sulphur into a beaker of cold water and examine the product. How could you discover whether the change was due to chemical action on the water or not? Set the product aside and examine it after a day or two (?).

In what substance have you found roll sulphur soluble? Find out whether this specimen is completely soluble in the same substance.

*c.* Rub a pinch of sulphur with a globule of mercury [Store-room] in a mortar (?). Recall cases of the union of sulphur with metals which you have met previously.

*d.* Make a thin paste of flowers of sulphur and distilled water by rubbing them together in a mortar, spread it evenly over the inside of a carefully cleaned, narrow-mouthed bottle. Allow the bottle to stand in your desk for several days. If the sulphur dries, repeat the moistening as often as is necessary. Examine the mass. Notice its odor (?) and hold a rod dipped in ammonium hydroxide solution over it (?). If it has dried up again, mix with 2-3 c.c. of water. Then filter, using a very small filter paper, and test the filtrate with neutral litmus paper (?) and with barium chloride solution (?). The result of the latter test may be slow in appearing.

*e.* [Hood.] Place a few particles of sulphur on the inverted lid of a porcelain crucible and heat strongly (?). Notice the odor of the gas and apply the ammonia test (?). Contrast the results of *d* and *e*,—the effects of oxidizing sulphur in presence and in absence of water.

#### 2. HYDROGEN SULPHIDE [Hood].

*a.* Place a little ferrous sulphide in a test-tube. Add some water for the purpose of dilution and then concentrated hydrochloric acid until a moderately rapid evolution of gas ensues (?). Notice the odor of the gas. Explain this action







with the help of the ionic theory, noting the extent to which hydrogen sulphide is ionized (Appendix).

Use a Kipp's apparatus and wash bottle containing water for obtaining the gas required in **2**, *b*, **3**, and **4**.

*b*. When the air has been displaced, attach a nozzle and set fire to the gas. What are the products of its combustion?

Hold a porcelain dish in the flame (?). What substance is shown by this observation to exist free in the interior of the flame? What other free body do you infer must be present in the same region? From these facts you may infer that the action takes place in two stages (?). What light do these facts throw on the difficulty in making the compound by direct union of the elements?

*c*. For comparison with **2**, *a*, treat two portions of powdered [Iron mortar] ferrous sulphide in separate test-tubes, one with concentrated sulphuric acid in the cold (?) and then in the heat (?), the other with dilute sulphuric acid (?).

What physical component is present in large amount in the concentrated acid, while in dilute sulphuric acid it is almost wanting? Can you explain the difference in the result on this ground (*cf.* p. 54, **8**, *b*)?

### 3. PROPERTIES OF HYDROGEN SULPHIDE: I [Hood].

*a*. Prepare an aqueous solution of the gas in a carefully cleaned test-tube, and expose the greater part of it to the air for some time in an open narrow-mouthed bottle (?). Preserve the rest for use in **4**, *g*.

Place the following substances in test-tubes and pass a vigorous stream of the gas through each for a few moments. This gives the same result as adding an aqueous solution of the gas. Study each action carefully. In explaining the actions in **3**, *c*, *d*, and *e* and in **4**, remember that hydrogen sulphide is somewhat ionized and that some sulphides give even less sulphidion under these conditions than it does.

*b*. Concentrated sulphuric acid (?). What substance might be used for drying the gas, and which substances commonly used for drying would be unsuitable?

*c*. Acidified potassium dichromate solution (?). ["Acidified" generally means that a dilute acid has been added until the solution has an acid reaction toward litmus. Use here a considerable excess of sulphuric or hydrochloric acid, and do not forget that excess of the acid is present.] Two kinds of ionic change occur here (*cf.*, p. 55, **9**).

*d*. Diluted and acidified potassium permanganate solution (?).

e. Ferric chloride solution (?). Saturate (test ?) the solution with the gas. Filter and add potassium ferricyanide solution to the filtrate (?).

The significance of the result will be brought out by taking ferric chloride and ferrous sulphate solutions in separate test-tubes and adding the ferricyanide to each (?). Formulate these two actions ionically.

Can you now state what the action of hydrogen sulphide was? How, precisely, were the ions of iron changed (*cf.* p. 55, 9)?

Recall the action of hydrogen sulphide solution on iodine (p. 40, 5, *b*). What general property of hydrogen sulphide do these experiments illustrate?

4. PROPERTIES OF HYDROGEN SULPHIDE: II. SULPHIDES OF METALS [Hood]. Dilute a few drops of each of the following solutions with five to ten times their volume of water, pass hydrogen sulphide to saturation (test ?) into each, and note the results (*cf.* 3, *a*, second par.):

a. Cupric sulphate.

b. Cadmium sulphate.

c. Lead acetate.

d. Zinc acetate.

e. Barium chloride.

All these actions are reversible when a sufficient concentration of acid (*i.e.*, hydron) is present in excess. In chemical analysis those of the above actions which are reversed by a rather small concentration of acid (*i.e.*, dilute acid), and those of them which require a high concentration, are put into two separate classes. There are also sulphides which are almost completely decomposed (and, in any case, completely dissolved) by a mere equivalent of acid.

Add pure dilute hydrochloric acid to the contents of each of the above test-tubes, agitate, and observe the effect. Classify these metals according as their sulphides seem to belong to one or other of these three classes.

[Advanced students.] Give the explanation of these facts in accordance with the theory of ionization.

f. Add sodium carbonate solution to lead nitrate solution in a test-tube, filter, and expose the paper with its precipitate to hydrogen sulphide gas (?).

g. Test the rest of the aqueous solution of hydrogen sulphide from 3, *a* with litmus paper (?).

h. Pass a stream of the gas into a few c.c. of sodium







hydroxide solution in a test-tube until the solution is perfectly saturated (test ?) (?). Test the solution with litmus paper and divide into three parts.

To the first add dilute hydrochloric acid (?).

To the second add a little powdered roll sulphur and shake from time to time (?). After half an hour, or when the solution has become very yellow in color, filter, and acidify the filtrate with dilute hydrochloric acid (?). What did the yellow solution contain [R]? Recall an experiment with iodine

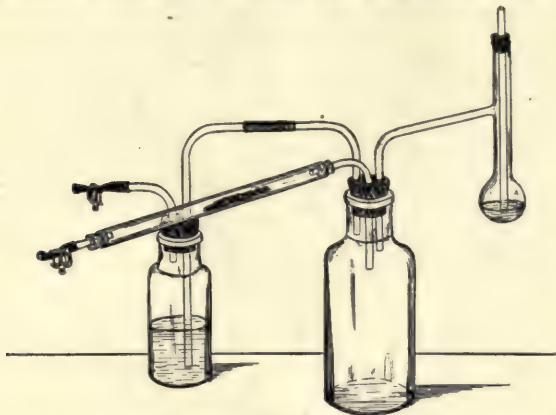


Fig. 18.

which resembles this action of sulphur on sodium sulphide. Filter, wash the precipitate with water, dry it, and test its solubility in carbon disulphide and its combustibility.

Leave the third portion in a partially closed bottle for several days (?). After a change in its appearance is plainly perceptible add dilute hydrochloric acid in excess (?). Explain.

##### 5. SULPHURIC ACID [Two students working together].

a. Obtain a distilling flask, a safety bottle with rubber connections, a rather wide-mouthed 1-liter bottle, and a Chapman pump from the store-room [Temp. order]. Fit the large bottle as in Fig. 18. Charge the hard glass tube with about 10 g. of granular pyrite. Place in the distilling flask about 10 c.c. of pure concentrated nitric acid [Side-shelf]. The safety bottle, half filled with water to show the rate at which air is being drawn through the apparatus, is attached to the water pump. The total air admitted is regulated by the screw clamp near the pump, while the proportions which pass over the pyrite and carry over the nitric acid vapor respect-

ively are regulated by pinching one of the tubes with the finger and thumb.

First heat the pyrite in a very gentle stream of air until the sulphur burns. Then warm the nitric acid and divert part of the air current so that it may carry over a little of the vapor of the acid; heat the pyrite strongly and continuously. Repeat the introduction of air laden with nitric acid at intervals, by pinching the tube admitting air to the pyrite-burner, whenever the disappearance of the red fumes in the bottle shows that a further supply is needed.

After a crust of crystals (?) has formed in the bottle (there may be considerable delay before crystallization starts) remove the attachments and blow the gases from the interior by means of the bellows. If crystallization fails to begin after a reasonable time (note that an interaction even between the molecules of gases may be slow, in spite of the completeness of the mixing), the cause is either the introduction of too much water along with the nitric acid, or the high temperature produced by chemical actions taking place in the bottle. Removing the attachments and cooling the bottle in a stream of water frequently brings it about.

Add 4-5 c.c. of water and wash down the crystals with it. Describe all that happens. If more of the product is required, the apparatus may be connected up again and a further supply of sulphur dioxide drawn into the bottle and subsequently more nitric acid vapor can be added. Finally any remaining crystals may be decomposed by water.

Filter the liquid in the bottle through a very small filter paper into a dish, rinsing the bottle with 2-3 c.c. of water, and evaporate on the sand bath [Hood] until the liquid begins to fume strongly (?). This will remove any nitric or nitrous acid that it may contain. Use the result for *b*.

*b*. Dilute the product from *a* by adding it to 2-3 volumes of water. Test the solution with litmus paper (?). In one-third of it place a piece of zinc or iron (?). To a small part add barium chloride solution (?). With the remainder make marks on a piece of paper by means of a match dipped in the liquid. Put the match and the paper on a radiator to dry (?).

*c*. [Hood.] Take 2-3 c.c. of concentrated sulphuric acid in a test-tube. Suspend a thermometer so that the bulb is completely immersed in the acid. Heat the contents of the tube by means of a small flame and note the temperature at which any effect (?) is observed and that at which it is conspicuous. [CAUTION: During the heating remember that, if the tube should crack, the hot acid may splash on the clothes and





hands and produce severe burns. Exercise proper caution. Be careful not to wash out this tube until the acid has cooled.]

**6. SULPHATES.** Place some ferric sulphate in a hard glass tube, or porcelain crucible, and heat strongly with the blast-lamp, continuing the heating after all the water has been driven off [R] (?). Relate this result to that in 5, *c*. Recall action of heat on dehydrated gypsum (p. 26, 3, *d*). Classify sulphates in accordance with this distinction [R].

#### 7. SULPHUR DIOXIDE.

*a*. Heat a piece of sulphur and a piece of charcoal with concentrated sulphuric acid in separate test-tubes. Notice the odor of the vapor. What property of sulphuric acid prevails in this case? Recall p. 57, 2, *c*, and 3, *b*, p. 37, 2, *a*, and p. 22 1, *e*, and compare.

Give a list of the properties of sulphuric acid which have been illustrated in 5, *b* and *c*, and 7.

*b*. [Hood.] Prepare a flask fitted like that in Fig. 10 and two gas washing bottles like the one in Fig. 12. If 8 is omitted, the second bottle containing the drying agent (see below) will not be required. Twist up some copper turnings [Be careful not to cut the fingers] into bunches, place them in the flask, and add 10–15 c.c. of concentrated sulphuric acid. Attach the first of the two wash bottles in reversed position, with the short tube next the flask and leave it empty to act as a safety bottle (?). Put an inch or so of concentrated sulphuric acid in the second. See that the apparatus is air-tight.

Heat the flask and contents by means of a sand bath. Leave the cork out at first and suspend in the acid a thermometer. Note the temperatures at which chemical action becomes perceptible (?) and at which it is conspicuous (?). Relate this result to the temperature found in 5, *c*. Why cannot dilute sulphuric acid be used? Connect the apparatus and continue heating to obtain the gas needed in 8 and 9.

Note the appearance of the contents of the flask as the action progresses, and account for it (?). In this connection read 9, *f*.

**8. WEIGHT OF A LITER OF SULPHUR DIOXIDE [Quant. Hood.]** Clean and dry a 250 c.c. flask and provide it with a tightly fitting cork. Weigh the flask and cork. This gives the weight of the flask filled with air. Now fill it completely with sulphur dioxide, by downward displacement of air, cork and weigh again. To insure its being full, repeat this operation till no increase in weight occurs. Finally, allow the gas to escape, and determine its volume by filling the flask with



water up to the cork and weighing again. Observe the temperature and pressure of the atmosphere.

To obtain the weight of the empty flask and its cork, subtract from the weight of the vessel filled with air the weight, under the observed conditions, of a volume of air equal to its content (1 liter pure dry air weighs 1.293 g. under normal conditions).

The difference between this corrected weight and that of the flask filled with sulphur dioxide is the weight of the latter. Reduce the volume of the gas to normal conditions and calculate the weight of 1 liter (?) and of 22.39 liters (?).

Enumerate carefully all the sources of error to which you would expect this way of determining the density of a gas to be liable. In doing this, consider each detail of the operation very critically.

### 9. SULPHUROUS ACID [Hood].

*a.* Pass the gas, prepared as in 7, *b*, for a few minutes into a test-tube full of water (?). Test the solution with litmus paper [R] (?). The result shows what is present in the aqueous solution of the gas. This product is to be regarded as the active substance in the following paragraphs.

*b.* To one-half of the liquid add barium chloride solution (?) and excess of pure hydrochloric acid (?). Is the action of barium chloride on the substance recognized in *a* reversible or not?

Then add bromine water to the same portion (?). What is the precipitate [R] (?) and how was it formed?

*c.* Expose the rest of the solution to the air in a beaker for a day or two, testing a few drops with pure hydrochloric acid and barium chloride solution from time to time (?). Relate the result to those in 1, *d* and *e* and 9, *b*.

*d.* Pass a stream of gas into test-tubes containing solutions of potassium dichromate (?) and potassium permanganate (?), each acidified (*cf.* p. 57, 3, *c*), with dilute sulphuric acid, until no further change is observed [R].

*e.* Collect a bottleful of the gas by downward displacement of air and put in it some moist litmus paper and some grass (?).

*f.* Allow the flask in which the sulphur dioxide was prepared to remain over night, examine and describe the appearance of all its contents carefully. Then, if there is any solid matter in the bottom, pour away the liquid and, after renewed, minute examination of the solid, throw some of the latter into a beaker of water. (If there is no solid, pour part of the







liquid into a large amount of water.) What are the physical properties of the solid? What do you observe when it dissolves in water? Explain.

### 10. SULPHITES.

*a.* Add any dilute mineral acid to sodium sulphite or sodium bisulphite (?).

*b.* Heat strongly [Blast-lamp] about a gram of each of these salts separately in dry test-tubes until no further change is observed (?). In each case begin to heat cautiously and hold the tube in a horizontal position to prevent cracking by condensed moisture.

Add dilute hydrochloric acid to each result (?). If free sulphur is observed, account for its formation.

### 11. THIOSULPHATES.

*a.* How are these salts prepared [R]? Take some diluted sodium thiosulphate solution and add any dilute mineral acid. What appears after a time? Note the odor.

Heat strongly a small quantity of sodium thiosulphate in a dry test-tube, using the same precautions as in 10, *b*, until no further change is observed. Observe the effect and notice the odor. Add dilute hydrochloric acid to the result (?).

**12. REDUCTION OF SULPHUR COMPOUNDS.** Mix a pinch of any salt of a sulphur acid with an equal amount of anhydrous sodium carbonate. Slightly char the end of a match and rub the charred part, which should be about an inch in length, with a heated crystal of sodium carbonate [Instructions]. Moisten the above mixture with water, place some of it on the end of the match, and heat in the reducing region of a small Bunsen flame. Put the result on a clean silver coin lying in a watch glass and moisten with one drop of water (?). Then add some dilute mineral acid and notice the odor (?). This is a test for sulphur in any form of combination.

## CHAPTER XIV.

### THE ACTIVITY OF ACIDS MEASURED CHEMICALLY.

1. ESTIMATION OF THE RELATIVE ACTIVITY OF ACIDS. The relative "strength" or activity of acids (or bases) can only be measured when the conditions for the acids compared are the same. When the acids are not on the same footing, as in the action of sulphuric acid on common salt, giving hydrochloric acid in the absence of much water (p. 32, 3, *d*), the fact that the volatile acid is almost entirely displaced does not give any information about the relative "strength" of the two acids. The following experiment illustrates the simplest of the four or five methods of comparing the activity of acids.

*a.* When methyl acetate is mixed with water, it undergoes hydrolysis very slowly, acetic acid and methyl alcohol being formed:  $\text{CH}_3\cdot\text{C}_2\text{H}_3\text{O}_2 + \text{H}_2\text{O} \rightleftharpoons \text{CH}_3\text{OH} + \text{HC}_2\text{H}_3\text{O}_2$ . Add about 1 c.c. of methyl acetate to 10 c.c. of distilled water in a test-tube, test with neutral litmus paper (?), and cork up and label the mixture. After several days, test once more with litmus (?).

This action of water is found to be greatly hastened by the addition of free acids, although the acids remain themselves unchanged by the process. Equivalent quantities (?) of different acids show very different accelerating powers toward this reaction. The order in which they are placed by measurement of this particular form of activity, however, is the same as that into which they fall when compared by any of the other methods. The extent to which the change has taken place can be measured at any moment by titration with alkali. The quantity of acid which was present at starting being known, the quantity found is the same acid plus the acetic acid set free by the progress of hydrolysis. Subtraction gives the quantity of the latter, and this quantity is a measure of the activity of the accelerating acid. The "strengths" of hydrochloric and sulphuric acids are compared in *b* by this method.

*b.* [Two students work together.] Procure two 20 c.c. stoppered graduated flasks and a 10-c.c. and a 1 c.c. pipette [Temp. order]. Mark the flasks so as to be able to distinguish them, and into one measure exactly 10 c.c. of normal (?) hydrochloric acid, and into the other 10 c.c. of normal (?)







sulphuric acid. Put exactly 1 c.c. of methyl acetate into each, and fill both flasks with distilled water up to the 20 c.c. mark at once (why at once?). Stopper the flasks tightly, mix the contents, and suspend both so that their necks are just above the water in a large bath heated to about  $45^{\circ}$ . If the bath is fairly large, further external heating will not be necessary. Otherwise, maintain the temperature by means of a small flame. In accurate work the temperature must be kept constant within  $0.1^{\circ}$  during the experiment by means of a thermostat. Allow the flasks to remain in this position for half an hour.<sup>1</sup>

While this is going on make some normal (or approximately normal, = 4 per cent.) sodium hydroxide and fill a burette with it. Take fresh portions of 10 c.c. of each of the acids and titrate them with the alkali, using two drops of phenolphthalein as an indicator. Record the results. These numbers measure the amount of mineral acid at starting in each flask.

When the above time has elapsed, remove both flasks from the bath, transfer the contents of each to a separate beaker, rinsing out the flasks with distilled water. Add two drops of phenolphthalein to each portion and titrate with the solution of sodium hydroxide used before. The difference between the volumes of alkali required for the neutralization of 10 c.c. of each acid with and without methyl acetate represents the amount of sodium hydroxide required to neutralize the acetic acid liberated in the hydrolysis. The two values obtained are functions of the activity of the acids. Which acid is more active? What, according to the theory of ionization, is really measured in these experiments? What does the result of *a* show water to be?

[Advanced students]. To learn the method of handling the results of measurements like this, so as to get a numerical expression for the relative activity of acids, some work on physical chemistry must be consulted.

<sup>1</sup> Instead of graduated flasks common flasks with cork stoppers may be used. Measure into each flask 10 c.c. of acid and then, with the same pipette, 10 c.c. of water. Add the 1 c.c. of methyl acetate, shake, and proceed as directed. Another action which may be adapted to the measurement of the relative activity of acids occurs in Chap. XXII, 2, *a* (p. 96).

## CHAPTER XV.

### OXIDES AND OXYGEN ACIDS OF NITROGEN.

#### 1. PRELIMINARY EXPERIMENTS.

*a.* Nitric acid [Hood]. Place a few grams of powdered sodium nitrate in a dry retort [Temp. order] or distilling flask, cover it with concentrated sulphuric acid, wait till the substances are thoroughly mixed, and then distil slowly. Use the sand bath as a means of heating and catch the distillate in a flask partly surrounded by water. [CAUTION: Use the greatest care in handling this liquid, as spilling it upon the hands may lead to very serious wounds.]

Describe the physical properties of nitric acid. Determine its boiling point by boiling it in a distilling flask and arranging a thermometer so that it is immersed in the vapor. Notice the color of the acid. Blow some air through the acid (?). To what was the color due? By what action was the colored substance produced? What property of nitric acid does this indicate?

*b.* Invert a jar of water in the pneumatic trough and half fill it with oxygen from the iron cylinder. Dilute the nitric acid prepared above with an equal volume of water in a small flask, fitted as in Fig. 10, add some copper turnings, and, after the air has been displaced, bubble the gas very slowly into the oxygen and agitate continually with water. Notice all the results.

Base a test for nitric acid on this experiment; also tests for oxygen and for nitric oxide.

*c.* Using these results, devise a way of determining the proportion of oxygen to residual gas in the air and test its accuracy by doing the experiment.

*d.* Action of heat on nitrates. Heat sodium nitrate in a hard glass test-tube (?). Use the blast-lamp, if necessary. Test the escaping gas for oxygen. When gas ceases to be evolved, preserve the residue for use in 5, *a*. The result is typical of the behavior of nitrates of alkali metals.

Heat 8-10 g. of powdered lead nitrate in a hard glass test-tube and conduct the gases into concentrated (50 per cent.; make this solution) sodium hydroxide solution in a test-





tube. Continue heating until gas is no longer evolved. What is the residue in the hard glass tube? The result is typical of the behavior of nitrates of heavy metals. Is all the gas absorbed by sodium hydroxide? If not, test the escaping gas for oxygen. Keep the sodium hydroxide solution for use in 5, *b*.

Cautiously heat some ammonium nitrate [Care] and collect the gas in a bottle over water. The result is peculiar to ammonium nitrate. Ignite a little red phosphorus in a deflagrating spoon and plunge it into the gas (?). What other gas is suggested? Suggest a test that would enable you to distinguish between the gases (?). What change in volume, if any, will accompany this combustion?

Classify nitrates in accordance with their behavior when heated.

## 2. PRINCIPLES INVOLVED IN MAKING NITRIC ACID.

*a*. Was nitric acid formed on mixing sodium nitrate and sulphuric acid before distillation began? Solve this question by mixing the materials (using finely powdered sodium nitrate), adding a very little water [Caution], agitating for a minute or so, and trying the test in 1, *b*.

*b*. Will a nitrate alone behave in the same way toward copper? Try sodium nitrate solution and copper (?).

*c*. Will other acids behave like sulphuric acid? Try phosphoric acid with powdered sodium nitrate, as in *a*. In view of the result, what should you expect to pass over if the mixture with phosphoric acid were distilled?

*d*. Is the action in *a* reversible? To answer the question take a few c.c. of concentrated sodium hydrogen sulphate solution and add an equal or greater volume of pure [Side-shelf] concentrated nitric acid. Cool the mixture in a stream of water and stir with a glass rod (?). [Failure may be due to the solution of the salt not being sufficiently concentrated.] Examine the result with a lens. What is formed? If the action is reversible, write the equation so as to show the existence of an equilibrium. What enabled us to obtain a large yield of nitric acid in 1, *a* in spite of this?

Give other instances of reversible actions.

## 3. PROPERTIES OF NITRIC ACID.

*a*. Is its solution an acid? Prove experimentally.

*b*. Recall the behavior of concentrated and of dilute sulphuric acid toward metals, and of the former toward non-metals.



Try the action of (1) magnesium and (2) zinc separately either on concentrated or on dilute nitric acid, and that of both on (3) copper. Explore the whole action thoroughly in each case as follows:

Place the metal in a side-neck test-tube (or 100 c.c. flask), furnished with a cork, provided with a dropping-funnel, and fitted with a delivery tube. Add the acid, and collect the gas over water after the air has been displaced from the apparatus. Observe whether nitrogen tetroxide is formed (?). Devise a way of separating nitric oxide from hydrogen, in case the gas collected should contain any of the latter. (Why can you not hope to recognize the hydrogen by its combustibility without separating it?). Examine the gas for each of these substances. If ammonia is formed by complete reduction of the nitric acid, where will it be found, and in what condition? (Consider all the circumstances carefully or you will answer wrongly.) Test for its presence (?). Isolate the form of combination which the metal has assumed, by evaporating the solution to dryness on the steam bath [Hood], and determine its nature (?). In view of the action of nitrogen tetroxide on water (1, *b*), explain the result you have observed when it is the diluted acid that is used with a metal. Conversely, the result with concentrated acid is explained by 4, *a*.

*c.* Try the action of tin on concentrated nitric acid (?). When the action has exhausted itself, add water and boil. Filter through a small filter paper, wash the precipitate with water, and see whether a neutral filtrate can be obtained (?). Explain what you observe. Devise a way of showing whether the remaining solid is a nitrate or not, and try it (?).

*d.* Try the action of sulphur on concentrated, boiling nitric acid (pure). Is there any evidence of action? If so, find out what form of combination the sulphur has assumed (?). Dilute the solution with water before adding any reagent.

What property of nitric acid seems to be most prominent? What points of resemblance and of difference does nitric acid show when compared with sulphuric acid?

*e.* Dip a piece of wool in concentrated nitric acid (?). The yellow coloring matter produced is xanthoproteic acid.

Give the three chemical properties which you have found to characterize nitric acid.

*f. Aqua regia.* Add concentrated hydrochloric acid to nitric acid, warm, and notice the appearance and odor (?). When gold and platinum (the same is true of other metals *a fortiori*) are placed in *aqua regia* they are dissolved and chlorides are formed. These metals are insoluble in dilute







acids (see table of solution tensions, p. 54, 8). Explain their solubility in this mixture. What other reagents would dissolve them on the same principle (p. 30, 1, *a*)? Which of the three properties of nitric acid is here brought into play?

#### 4. NITRIC OXIDE.

*a.* Prepare the gas by the action of copper on slightly diluted nitric acid in a side-neck test-tube. Pass the gas through warm concentrated nitric acid (?). What change is produced in the gas by the acid? Use this result to account for the product of the action of a metal on concentrated, as distinguished from dilute, nitric acid (*cf.* 3, *b*).

*b.* Prepare a solution of ferrous ammonium sulphate and divide into four parts. [The double salt is supplied in the laboratory instead of ferrous sulphate simply because it keeps better. The ammonium sulphate may be disregarded here.]

Pass the gas, which must be colorless, through one part(?).

*c.* Acidify the second portion of the ferrous ammonium sulphate solution with sulphuric acid, heat to boiling, and add nitric acid drop by drop (mix after each drop), until there is no further action. To ascertain what the solution contains, add ammonium hydroxide(?). Obtain the information necessary to explain the action by adding ammonium hydroxide to the third portion of the ferrous solution (?) and to ferric chloride solution (?). What change did the nitric acid produce in the diferrion (*cf.* p. 55, 9)?

*d.* Delicate test for nitric acid, or a nitrate. Add a very little sodium nitrate solution to 2-3 c.c. of the ferrous solution in a test-tube and pour concentrated sulphuric acid steadily down the side of the tube so that it may form a layer at the bottom. Notice the brown ring and explain with the help of the above results.

#### 5. NITROUS ACID.

*a.* Dissolve in a few drops of water the residue from heating sodium nitrate in 1, *d*. If it is necessary to repeat, melt the nitrate with a piece of lead in a crucible and stir with a file (?). This gives the same product more easily (why?). To the highly concentrated aqueous extract, add dilute sulphuric acid. Contrast the action with that on a nitrate (?).

*b.* Examine the solution, obtained by passing the gases from lead nitrate into sodium hydroxide in 1, *d*, for nitrite, by acidifying (test?) with the minimum amount of concentrated sulphuric acid (?). Add a drop of this mixture to some starch emulsion containing a drop or two of potassium iodide

solution (?). To a small portion of the same mixture add a drop of diluted potassium permanganate solution (?). What substance is shown to be present by these three reactions? Try these three tests with a concentrated solution of sodium nitrite (?).

Boil the remainder of the acidified mixture until red fumes cease to be evolved. Then insert a piece of copper (?). What substance is shown to be present by this reaction? Combining these results, what do you infer was the action of nitrogen tetroxide on sodium hydroxide?







## CHAPTER XVI.

### PHOSPHORUS, ARSENIC, ANTIMONY, BISMUTH.

1. PHOSPHORUS. What difference in behavior do the two allotropic modifications of phosphorus exhibit [R]. What is formed when phosphorus unites directly with oxygen? Answer from previous knowledge.

2. PHOSPHINE [Hood]. Place a small piece of calcium phosphide in water or dilute hydrochloric acid in a beaker (?). Relate this to ways of making other hydrides of non-metals (*e. g.*, ammonia, hydrogen sulphide, and acetylene) from compounds of the non-metal with a metal. In what ways does phosphine differ from ammonia [R]?

3. HALIDES OF PHOSPHORUS [Hood]. The actions of water on the tribromide and tri-iodide (p. 39, 3), have already been noticed.

a. Place 1 c.c. of phosphorus trichloride [Store-room] in a test-tube (?). Blow the breath over the mouth of the tube (?). Add water a drop at a time (?). Add more water and boil. Pass hydrogen sulphide through the solution (?).

b. Place a few small granules of phosphorus pentachloride on a watch glass. Blow the breath over it (?). Throw it into some water in a test-tube (?) and boil. To part of the solution add excess of silver nitrate solution (?). Filter. What remains on the filter? To the filtrate add ammonium hydroxide drop by drop (?). The nature of this precipitate is learned in 4, a.

#### 4. PHOSPHORIC ACID.

a. Heat a very little red phosphorus with excess of slightly diluted nitric acid (?). When the action has ceased, filter, if necessary, and drive off the water and excess of nitric acid from the filtrate on the steam bath (?). Re-dissolve in water the sirup which remains. Test the solution with litmus paper (?). Add silver nitrate solution. The precipitate is silver orthophosphate (a black precipitate, due to the presence of phosphorous acid, may sometimes be formed). What was the acid [R]?

b. Throw a pinch of phosphorus pentoxide, in minute portions at a time, into cold distilled water in a test-tube (?).

Allow the solution to stand for a few minutes, or until it becomes clear. Add silver nitrate solution (?). What acid was formed [R]?

### 5. PHOSPHATES.

*a.* Test some sodium phosphate (secondary sodium orthophosphate) solution with neutral litmus paper (?). Add silver nitrate solution (?) until the precipitation is complete. Test the filtrate from this with litmus again (?). Are acid salts always acid toward litmus? If not, explain why they are not.

Add excess of ammonium chloride and a little ammonium hydroxide solution to magnesium sulphate solution. Add some of this "magnesia mixture" to diluted sodium phosphate solution (?). Describe the precipitate.

*b.* Heat a little dry sodium phosphate in a crucible strongly [Blast-lamp] for twenty minutes, or until every part of the substance has been affected and no further change is observed (?). When the residue is cold, dissolve it in water and add silver nitrate solution (?). Contrast with the precipitate from unignited phosphate (5, *a*) and compare with that in 4, *b*.

*c.* Heat a little microcosmic salt strongly in a porcelain crucible, using the same precautions as above, and notice its behavior and odor (?). Dissolve the residue in water and add silver nitrate solution (?).

Make a bead of microcosmic salt on a platinum wire and fuse with it a single minute particle of cupric oxide (?).

What difference do your experiments show between the stabilities of sodium metaphosphate and sodium nitrate (p. 66, 1, *d*)?

### 6. ARSENIC.

*a.* Heat a particle of arsenic on a crucible lid (?). Notice its behavior and the odor. Do the same with a particle of realgar (?).

*b.* Boil a little (0.3 g.) powdered arsenic with excess of nitric acid. What evidence is there of action (*cf.* 4, *a*)? Preserve the solution for examination under 9. [Do 8 next and then return to 7.]

**7. ARSINE** [Hood. CARE! POISON!]. Arrange a side-neck test-tube with safety and delivery tubes and nozzle to generate and burn hydrogen. Place in it a piece of chemically pure zinc and add pure hydrochloric acid [Side-shelf]. When the air has been displaced [Care. Test?], light the gas and hold a





crucible lid in the flame (?). If there is no deposit, add a drop or so of the solution of arsenic trichloride (8, b), observe the appearance of the flame, and obtain a deposit on the crucible lid (?). What kind of chemical change takes place in the flame (*cf.* p. 57, 2, b)? Heat the tube, through which the gas passes to the nozzle, with a Bunsen flame (? Marsh's test). When these experiments are completed, fill the test-tube with water to stop the action.

Describe the appearance of the deposit (?).

Apply fresh bleaching powder solution to the deposit on the crucible lid by means of a glass rod (?).

What way of making arsine is identical in principle with that used for phosphine in 2 [R]?

How do ammonia and phosphine behave when heated [R]?

#### 8. ARSENIC TRIOXIDE (ARSENIOUS ANHYDRIDE).

a. Boil a pinch of the trioxide with sodium hydroxide solution (?). To what class of oxides does this one appear to belong?

b. Boil a pinch of the trioxide with concentrated hydrochloric acid (?). Dilute with a little water and keep part of the solution for 7. To what class of oxides does it appear now to belong?

Pass hydrogen sulphide from a Kipp's apparatus through the rest of the solution (?) until it is saturated (test ?). Recall result of 3, a and compare (?). Filter, reject the filtrate, and pour hot colorless or yellow ammonium sulphide solution on the precipitate [R] (?). Acidify (test ?) with hydrochloric acid the solution in ammonium sulphide which runs through (?).

c. Seal one end of a small piece of glass tubing. Mix a pinch of the trioxide with a little powdered wood charcoal. Place some of the mixture in the bottom of the tube, with a little fresh wood charcoal above, and heat strongly (?).

9. ARSENIC ACID. Neutralize the solution of arsenic in nitric acid (6, b) with ammonium hydroxide, avoiding excess. Divide into two parts. To the first add silver nitrate solution (?). To the second add "magnesia mixture" (?). Recall 5, a, and compare.

10. ANTIMONY. Behavior similar to that of arsenic.

11. STIBINE. Follow all directions in 7, using antimony trichloride solution in place of that of arsenic trichloride.

#### 12. ANTIMONY TRICHLORIDE.

a. Place some of the crystals in a test-tube and add a little water (?). Test the liquid with litmus paper (?). Add



more water, warm, and make a clear solution by adding small quantities of concentrated hydrochloric acid (?), agitating vigorously after each addition.

b. To half of this solution add a large amount of water [R] (?). What kind of action is this? Add concentrated hydrochloric acid again (?). How does this illustrate the influence on a reversible action of change in concentration of one factor? Write the equation which combines both actions.

How could you show that the molecules of the trichloride are only partly hydrolyzed by water, leaving a basic salt (with phosphorus trichloride the hydrolysis was complete)? What is the significance of this difference [R]?

c. Through the rest of the trichloride solution pass hydrogen sulphide from a Kipp's apparatus (?) to saturation (test ?). Filter; reject the filtrate, and pour hot yellow ammonium sulphide solution on the precipitate [R] (?). It may be better to scrape the precipitate into a beaker and boil it with the reagent. Acidify with excess of hydrochloric acid the solution in ammonium sulphide (?).

**13. ANTIMONY TRIOXIDE:** Heat some powdered antimony with concentrated nitric acid (?). How could you ascertain whether the result was a nitrate or not? How does this differ from the behavior of arsenic (6, b) [R]? Filter.

Boil one-half of the powder with hydrochloric acid (?). What kind of oxide does the result show it to be?

Boil the other half with sodium hydroxide solution (?). What kind of oxide does this show it to be?

**14. BISMUTH.** Prepare a match as in Chap. XIII, 12 (match test). Place on the end a moistened mixture of bismuth nitrate and anhydrous sodium carbonate and heat in the reducing region of a small Bunsen flame. Break up the charred match gently in water in the mortar, wash away the lighter particles, and examine the residue (?).

#### 15. SALTS OF BISMUTH.

a. Add water to some crystals of bismuth nitrate in a test-tube (?). Obtain a clear solution by adding small quantities of concentrated nitric acid.

b. To one-half of the solution add a large amount of water (?). Write the equation to show that an equilibrium exists.

If difficulty is found in obtaining a precipitate with water and the nitrate, make a clear solution, as in a, with the help of hydrochloric acid and then dilute with water.







c. Dilute the remainder of the solution from *a* with water and pass hydrogen sulphide through it (?). Filter and discard the filtrate. Treat the precipitate with warm yellow ammonium sulphide. Filter and acidify this filtrate. What is precipitated? Was the bismuth sulphide dissolved? Compare the result with that in 8, *b* and 12, *c*. How could you separate the sulphides of arsenic and antimony from that of bismuth?

## CHAPTER XVII.

### CARBON.

#### 1. CHARCOAL.

*a.* Place a small piece of charcoal in a test-tube half full of water (?). Now sink it, if necessary, with copper wire and boil the water for several minutes (?). When the whole has cooled, test once more the tendency of the charcoal to float (?). Explain.

*b.* Boil dilute solutions of litmus and indigo, separately, with powdered animal charcoal and filter each liquid (?). The activity of the charcoal is much increased by previous heating in a covered crucible.

*c.* In a hard glass test-tube mix intimately 2 g. of powdered cupric oxide with 1 g. of powdered wood charcoal and heat [Blast-lamp]. Pass the gases through lime water (?). Examine the residue by rubbing it in the mortar and washing away the lighter particles (?).

#### 2. CARBON DIOXIDE.

*a.* Fit up a generating flask with a safety tube and connect with two wash bottles containing water and concentrated sulphuric acid respectively (what is the use of each of these? The latter is unnecessary if 3 is omitted). Place in the flask some pieces of marble and pour upon them diluted hydrochloric acid. Collect the gas in three bottles by upward displacement of air.

*b.* Use the first to ascertain whether the gas is soluble in water or not.

Use the second to compare its weight with that of air. Use baryta water or lime water as a test.

Use the third to test its power of supporting combustion.

*c.* Lead the gas into a little sodium hydroxide solution in a test-tube until the solution is saturated (test ?). Let the solution dry spontaneously (first residue). Heat the dry residue (?) in a test-tube and determine what two things are given off.

To this residue after heating (second residue) add dilute hydrochloric acid until all action (?) ceases. Evaporate the solution on the steam bath and examine and taste this final residue (?).







Having recognized the products of the last action and taking into account the preceding observations, state what the nature of the second and first residues must have been. Write equations for all actions.

**3. MOLECULAR WEIGHT OF CARBON DIOXIDE [Quant.].** Determine the weight of 1 liter of the gas by the method used for sulphur dioxide (p. 61, 8), and calculate the weight of the gram-molecular volume at 0° and 760 mm. What further information must we have to enable us to determine the formula?

Use the quantitative results you obtained in the synthesis of carbon dioxide (p. 15, 1), along with this molecular weight, to calculate the weights of carbon and of oxygen in a molecular weight of the gas. What further steps are necessary in order to fix the atomic weight of carbon?

#### 4. CARBON MONOXIDE.

*a.* Heat about 10 g. of oxalic acid crystals with concentrated sulphuric acid in a generating flask and fill a bottle with the gas which is given off. Shake with lime water (?). With what substance should we wash the gas to remove the carbon dioxide? Arrange a wash bottle to purify the gas. Fill two bottles with the purified gas over water. Test one with lime water again (?). If the gas is pure, burn that in the other bottle, add lime water at once, close quickly and shake (?).

*b.* Devise a way of ascertaining roughly the relative volumes of the two gases generated in *a* and measure the proportion in a test-tube full of the mixed gases.

**5. MOLECULAR WEIGHT OF CARBON MONOXIDE [Quant.].** Arrange a 250 c.c. flask as in Fig. 3, using, however, a round-bottomed flask for the purpose. Make a mark on the neck at the bottom of the stopper, so as to be able to measure the exact content of the flask up to the stopper. Place 30 c.c. of water in the flask, remove the clip, which must be a strong one, and boil the water with a small flame for about five minutes, so as to drive out all the air. Close the rubber tube with the clip and remove the flame quickly, wipe the flask and allow it to cool. When it has assumed the temperature of the air, weigh the whole carefully, suspending the apparatus on the balance by a wire. Connect with the apparatus delivering pure carbon monoxide, and open the clip a very little so as to admit a slow stream of the gas. When the flask is full, close the clip, disconnect from the generating apparatus, open the clip for an instant to restore the pressure to that of the

atmosphere and weigh again. The gain in weight represents the weight of the carbon monoxide. Read the barometer and thermometer. Subtract from the barometric reading the aqueous tension at the observed temperature. Ascertain the volume of the flask by filling with water to the mark and weighing again.

Calculate the weight of 1 liter and of the gram-molecular volume of the gas at  $0^{\circ}$  and 760 mm.

To what class of gases would this method of determining the density and molecular weight be applicable? Why could not this method be used for carbon dioxide?

## 6. ACIDS.

a. Add some dilute sulphuric acid to sodium acetate and warm. Notice the odor. How could you obtain acetic acid? How is it manufactured [R. Organic chemistry]?

b. Take some acetic acid and test its reaction with litmus (?). Recall its action on iron (p. 22, 1, d). Add 1 g. of litharge (lead monoxide) to 2.5 c.c. of acetic acid and boil (?). Filter, if necessary, and set the clear solution aside to crystallize. Describe the product. What is its common name [R]?

7. ALCOHOL. Dissolve 20 g. of molasses in 150 c.c. of water and add a little yeast. Fill a flask to the base of the neck with the mixture, plug the mouth loosely with cotton, and set the whole aside for 3-4 days. At the end of this time warm the solution and test the gas which is given off for carbon dioxide [R].

Set up a condenser [Temp. order] and distilling flask (Fig. 16). Filter the liquid and distil off about 50 c.c., using an ordinary flask connected with the condenser by an L tube. Boil this portion in the distilling flask with a small flame and catch the part which passes over between  $80^{\circ}$  and  $93^{\circ}$ .

Notice the odor of the distillate (?). Test its reaction with neutral litmus paper (?). Use one drop to ascertain whether it burns. To the rest add a crystal of iodine and enough sodium hydroxide solution to dissolve it. Shake vigorously and do not add more alkali than is absolutely necessary. Warm the solution and then cool it (?). This is the iodoform test.

8. ESTERS. Why is the name "ethereal salts," commonly given to these substances, inconsistent with the definition of a salt [R]?

a. Dissolve about 1 g. of sodium acetate in a very little water, add a few drops of concentrated sulphuric acid and





two or three drops of alcohol Warm and notice the odor [R]. This is used as a test for acetic acid.

b. Place in a porcelain dish a piece of fat the size of a pea, and add 2 c.c. of alcohol and five drops of 50 per cent. sodium hydroxide solution. Stir constantly and boil very gently until the odor of alcohol is no longer perceptible, then stop. The alcohol is used as a common solvent for the fat and the alkali. What is the residue [R] ?

Dissolve the soap in hot water, cool, and to half of the solution add dilute hydrochloric acid and shake vigorously (?). Withdraw the floating coagulum by means of a glass rod, suspend it in water in a test-tube, add a few drops of sodium hydroxide, and heat until solution takes place. What do you conclude from its solubility in alkali?

To the other half of the soap solution add calcium chloride solution (?). Explain the action of hard water [R] on soap solution.

### 9. HYDROCARBONS.

a. Powder some fused sodium acetate and some sodium hydroxide and mix them intimately in approximately equal proportions. Then add a little powdered quicklime and iron filings (these are not necessary for the chemical action in itself), and mix again. Heat the mixture in a hard glass test-tube clamped in a horizontal position and fitted with a cork and delivery tube. Catch some of the gas in a bottle by displacement of water, and explode it with air (?). Burn the gas in a jet and note the degree of luminosity of the flame (?). Consider how you will ascertain what it forms in burning, and try experiments to settle the matter.

b. Fit a 250 c.c. flask with a doubly bored cork, through which pass a dropping-funnel and L tube, and connect with an empty bottle (why?) provided with a doubly bored cork through which pass an L tube and a tube drawn out to a nozzle. Place in the flask 5-10 g. of dry, broken glass. Clamp the flask upon the ring stand over a sand bath. When the whole apparatus is ready, and its airtightness has been ascertained (and not until then), put about 5 g. of phosphorus pentoxide into the flask and close it quickly (why?). Mix the pentoxide with the broken glass by shaking, and set the flask in position again. Introduce into the bulb of the dropping-funnel some alcohol. Finally heat the pentoxide and, when it has had time to reach 150-170°, admit the alcohol a drop or two at a time to the flask.

When the air in the apparatus has been displaced, fill a narrow-mouthed bottle, provided with a rubber or greased

glass stopper, with the gas (?) by downward displacement of air.

Add a drop of bromine [Hood] to the gas and replace the stopper instantly. Observe what happens, and, after a minute, open the bottle under water (?) [R].

Burn a jet of the gas and observe the degree of luminosity of the flame.

Why is the accepted formula for this gas preferred to the simplest? What volume of oxygen would be required to burn a volume of the gas completely? What would be the volumes of the products?







## CHAPTER XVIII.

### SILICON AND BORON.

1. **SILICA.** Mix 1 g. of finely powdered silica with 4-5 g. of anhydrous sodium carbonate. Make a small watch-spring spiral on the end of the platinum wire [Instructions] and, by alternately heating in the Bunsen flame or blast-lamp, and dipping in the mixture, obtain a large bead and heat it strongly till all action (?) seems to have ceased. Place the bead in a test-tube and make others by the same process. Dissolve the beads in a small amount of water. Add hydrochloric acid a drop at a time until the solution is strongly acid (?). Evaporate the solution to dryness on the sand bath (?). Treat the residue with warm water, wash the whole contents of the dish into a test-tube and examine (?).

2. **A SILICATE.** Mix dry potassium carbonate with anhydrous sodium carbonate in equal proportions in a mortar. Coil the platinum wire to watch-spring form. Mix a little powdered talc (is this soluble in water? What is its common name?) with 6-7 times as much of the "fusion mixture" and hold some of the result on the platinum wire in the flame of the blast-lamp till it is completely melted and all action (?) has ceased. Repeat till several beads are obtained. Treat the beads with boiling water in a test-tube until they are completely disintegrated. Filter through a small filter paper and wash the precipitate with water. Preserve this filter paper and precipitate for use later. Acidify the filtrate with concentrated hydrochloric acid and proceed as in 1.

Make a hole in the paper and wash the precipitate obtained above into a test-tube. Add dilute hydrochloric acid and warm (?). Filter, if necessary, and add ammonium hydroxide to alkaline reaction (?) The precipitate is aluminium hydroxide. Boil and filter. To the filtrate add a few drops of ammonium hydroxide, some ammonium chloride solution and some sodium phosphate solution and shake (?). Compare with Chap. XVI, 5, a (p. 72).

### 3. BORIC ACID.

a. Dissolve some borax in distilled water. Test this solution and a sample of the distilled water simultaneously with

neutral litmus paper, and, by comparing tints, determine whether the solution has any reaction (?).

Put two drops of the solution into a test-tube and dilute with water till the tube is two-thirds full. To the remainder add silver nitrate solution (?). Add silver nitrate solution to the very dilute solution also (?). The difference is more marked if the dilute solution is first warmed. For comparison, add silver nitrate solution to an exactly equally diluted sodium hydroxide solution (?). What conclusion do you draw in regard to the action of water on borax? Write the equation. Is the action reversible [R] ?

*b.* Make a strong solution of borax in boiling water in a test-tube. Add concentrated hydrochloric acid until the solution is strongly acid and set aside to cool (?). Filter, press out the mother liquor, and wash the crystals with a few drops of cold water. Dissolve in the minimum amount of boiling water and set aside again. Filter, and wash the crystals as before.

Dissolve part of the crystals in hot water and test the reaction of the solution with neutral litmus paper, using the same precaution as in *a* (?). What conclusion do you draw in regard to boric acid? Dip a strip of turmeric paper in the same solution, wrap it round the upper part of the test-tube and boil the solution until the paper is dry (?). Touch it with a glass rod dipped in sodium hydroxide solution (?). This is a test for boric acid.

Treat the rest of the crystals with cold sodium hydroxide solution (?). Explain the formation of boric acid and its solubility in bases according to the ionic theory.

*c.* Place on separate parts of a watch glass a drop of concentrated sulphuric acid, a drop of glycerine, and a very little powdered borax. Rub the end of a platinum wire in each of these. Bring the end of the wire slowly up to the outer edge near the bottom of a small Bunsen flame. How is the flame colored? This is a test for a borate.







## CHAPTER XIX.

### METALS OF THE ALKALIES.

#### 1. POTASSIUM HYDROXIDE.

*a.* Dissolve about 30 g. of potassium carbonate (what is the source of this salt?) in 200–300 c.c. of water in a large beaker and heat to boiling. Slake 15–20 g. of quicklime in a beaker (?), using heat if necessary to start the action, and make the product into a very thin paste with water. Add this gradually, and with constant stirring, to the boiling solution (?). Continue boiling for a few minutes. (Why are iron utensils exclusively employed in this operation when it is performed on a large scale [R]?) Let the solution settle and, when it is cold, decant the clear liquid. Use the solution in *b* and *c*.

What kind of hydroxides alone can be made by this method? Which hydroxides are of this kind [R]?

*b.* Alkalimetry. Find the strength of this solution by titration. To do this, place a carefully measured volume (about 10 c.c.) of the clear solution in a small flask. Dilute with about four times its volume of water, as the concentrated solution is apt to decompose the indicator. Fill a burette with “normal” hydrochloric acid. Add some phenolphthalein solution to the alkali and run in the acid cautiously until the red color just disappears. Notice the volume of acid used. One liter of the acid contains 36.5 g. of hydrogen chloride.

Calculate the weight of potassium hydroxide per liter, which your measurement shows to be contained in the alkaline solution made in 1 (?). Express this also in terms of a normal solution containing 56 g. per liter (for example, 28 g. per liter would be .5 normal).

*c.* Reactions of OH ions. Place very small quantities of the following solutions in separate test-tubes, dilute with water, and add some of the solution of potassium hydroxide to each; ferric chloride (?); cupric sulphate (?); mercuric chloride (?) [R]. Boil the contents of each test-tube (?).

What kind of hydroxides can be made by this method? Do any metals fail entirely to form hydroxides [R]?

**2. PREPARATION OF POTASSIUM NITRATE.** Dissolve 25 g. of sodium nitrate and 22 g. of potassium chloride in 50 c.c. of

water and evaporate to half the volume on the sand-bath. Decant the hot, clear liquid from the crystals and set it aside. Throw the crystals which appeared during boiling at once onto a filter (p. 31, note) and rapidly press out the rest of the mother liquor with a spatula. Examine the form of the crystals and ascertain what they are. (If they are too small, recrystallize a part slowly from water in a beaker in order to learn their form.) When the decanted liquid is cold, press the product on a filter likewise. Examine this set of crystals as before. [R. cf. figures in Roscoe and Schorlemmer.] Compare both with the original substances.

To understand the process study the solubilities of the substances concerned as they appear in the following table:

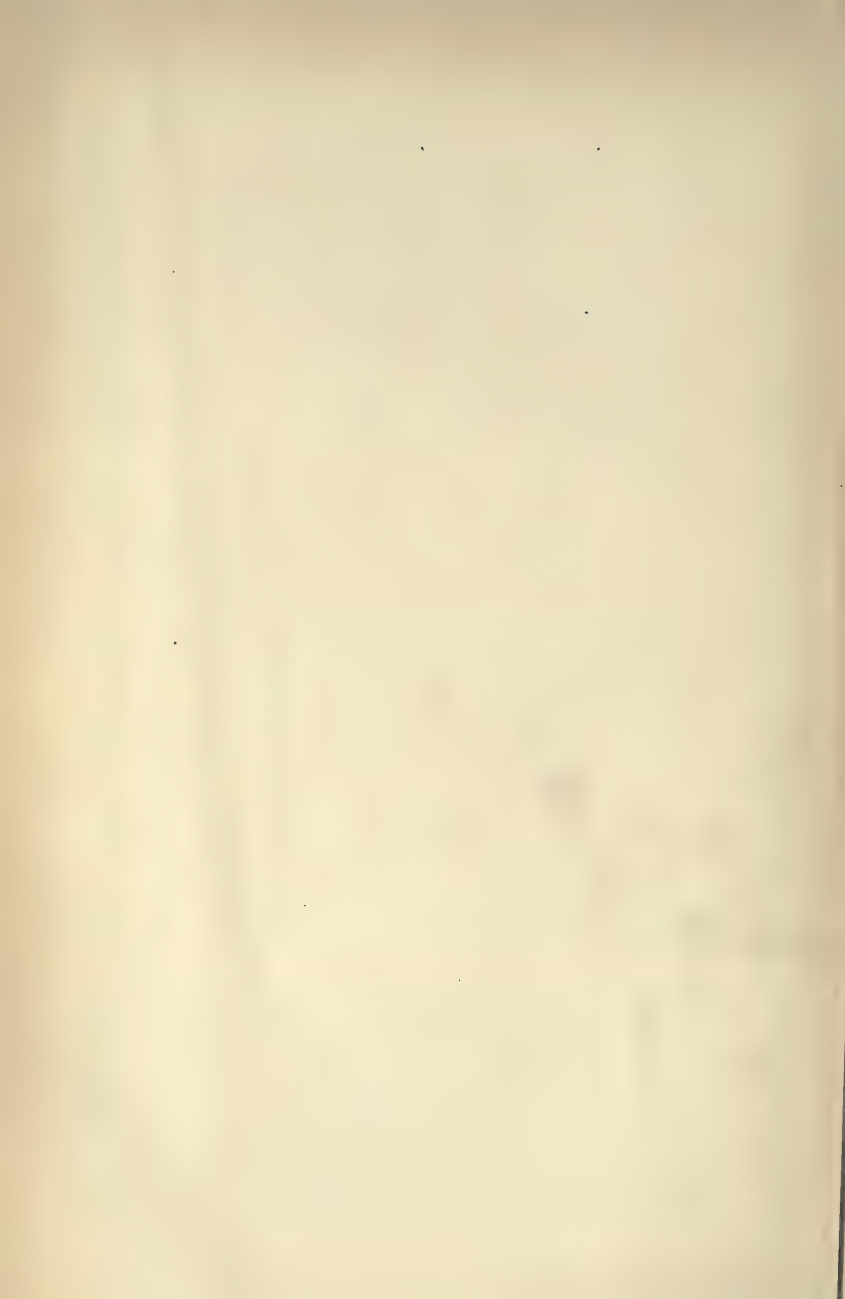
		Grams in 10 c.c. of water	
		10°	100°
Potassium nitrate	- - - - -	2.1	24.6
Sodium chloride	- - - - -	3.6	4.0
Potassium chloride	- - - - -	3.1	5.6
Sodium nitrate	- - - - -	8.1	18.0

Which of these substances will first be deposited from the boiling liquid? Ascertain by calculation how much of it (roughly) will be deposited at 100°, how much more will come out when the liquid cools, and how much will remain in the mother liquor. What other substance will be present in large quantity in the hot mother liquor, and how much of it must there be? How much of this product will be deposited when the liquid cools, and how much will be lost by remaining dissolved? What per cent of the possible yield may we expect to get? Dry your product, weigh it and calculate what per cent. was obtained.

Explain why purer potassium nitrate can be obtained by crystallizing the product once more from water. Whence the necessity for making the salt commercially by this method at all [R]?

**3. POTASSIUM CYANIDE [POISON].** How is this salt obtained? Place 2 c.c. of potassium cyanide solution in an evaporating dish, heat it, and add yellow ammonium sulphide solution until the color no longer disappears. Evaporate to complete dryness [Hood]. Dissolve a part of the residue with water, and add ferric chloride solution (?). A black precipitate (?) indicates that the heating was not sufficient. If this appears, heat the remainder once more and try the action of ferric chloride again. What property of the cyanides does the first part of this experiment illustrate [R]?





#### 4 REACTIONS OF POTASSIUM SALTS.

*a.* Heat a little solid potassium nitrate on a clean platinum wire. Notice the color of the flame and examine with the spectroscope. Make a diagram showing the position of the lines with reference to the D line, which, on account of the sodium present, is shown by all flames in the laboratory.

*b.* Add a strong solution of the nitrate (made by warming), to tartaric acid solution. Shake the mixture and cool in a stream of water (?) Note, also, the effect of rubbing the inside of the test-tube with a glass rod. Describe the product. Filter, press out the mother liquor, and wash the precipitate with a little alcohol (see note). Dry by pressing between filter papers. The nature of the product (?) may be inferred from the results of *c* and *d*.

*c.* Dissolve a little of the precipitate in warm water and test its reaction with litmus paper.

*d.* Place half the remainder in a test-tube and add sodium carbonate solution a drop at a time, mixing thoroughly, and noticing all that happens (?) If any change is observed, let the action go on until it is complete. Can you explain why evidence of the beginning of the action is so slow in making its appearance?

Add concentrated hydrochloric acid, a drop at a time, to the resulting solution (?). Stir vigorously with a glass rod at intervals. Finally try the effect of an excess of hydrochloric acid (?)

*e.* Heat the rest of the precipitate strongly in a porcelain crucible (?). Extract with hot water, filter, and add any acid to the filtrate (?). The ignition of all potassium or sodium salts of organic acids gives the same result.

*f.* To potassium chloride solution, add picric acid solution (?).

What is shown to be present in a solution when we get the tests in *b* and *f*?

*Note.*—Cut the filter-paper to circular form and use the smallest that will hold the precipitate. In washing, first let the mother liquor drain away completely, and then cover the contents of the funnel, including the whole paper (why?), completely with the washing material.

**5. SODIUM.** Recall the properties of the metal as you have met them in previous exercises. From previous knowledge compare its behavior with that of zinc and copper towards (*a*) air; (*b*) water; (*c*) acids. What mineral is the source of the metal and all its salts?



**6. SODIUM CARBONATE BY SOLVAY PROCESS.** Take 75 c.c. of ammonium hydroxide solution, diluted with 25 c.c. of water, dissolve in it 25 g. of powdered ammonium carbonate by shaking, and then saturate the solution completely with sodium chloride by prolonged agitation with finely powdered salt in a corked bottle. If common salt is employed, it should be washed with water before use. Decant the clear liquid into another bottle, fitted with cork and two tubes, one of which reaches to the bottom. Through the latter, pass in carbon dioxide from a Kipp's apparatus until the solution is saturated. This operation may occupy an hour or more. During the absorption of the carbon dioxide, the exit tube should be closed to prevent waste of the gas. Close the tubes with caps of rubber tubing plugged with glass rods [Instructions] and set aside over night (?). Filter off the deposit and dry by pressing between filter papers.

Dissolve in water a little of the solid, which must have ceased to smell of ammonia, and test the reaction of the solution with litmus (?).

To part of the solid add any dilute mineral acid (?).

Heat the rest in a test-tube clamped so that the mouth is inclined slightly downward, and ascertain what gases are evolved. When gas ceases to be given off, dissolve the cold residue in a very little water, test the reaction of the solution with litmus paper (?), and set it aside to crystallize in an open dish (?). Ascertain the effect of exposure in an open vessel (?) and the action of acids (?) on the the crystals.

The solubilities at 20° of the bicarbonate and carbonate in 100 parts of water are 9.6 and 92.8 respectively. Explain why the bicarbonate is made first and then the carbonate from it.

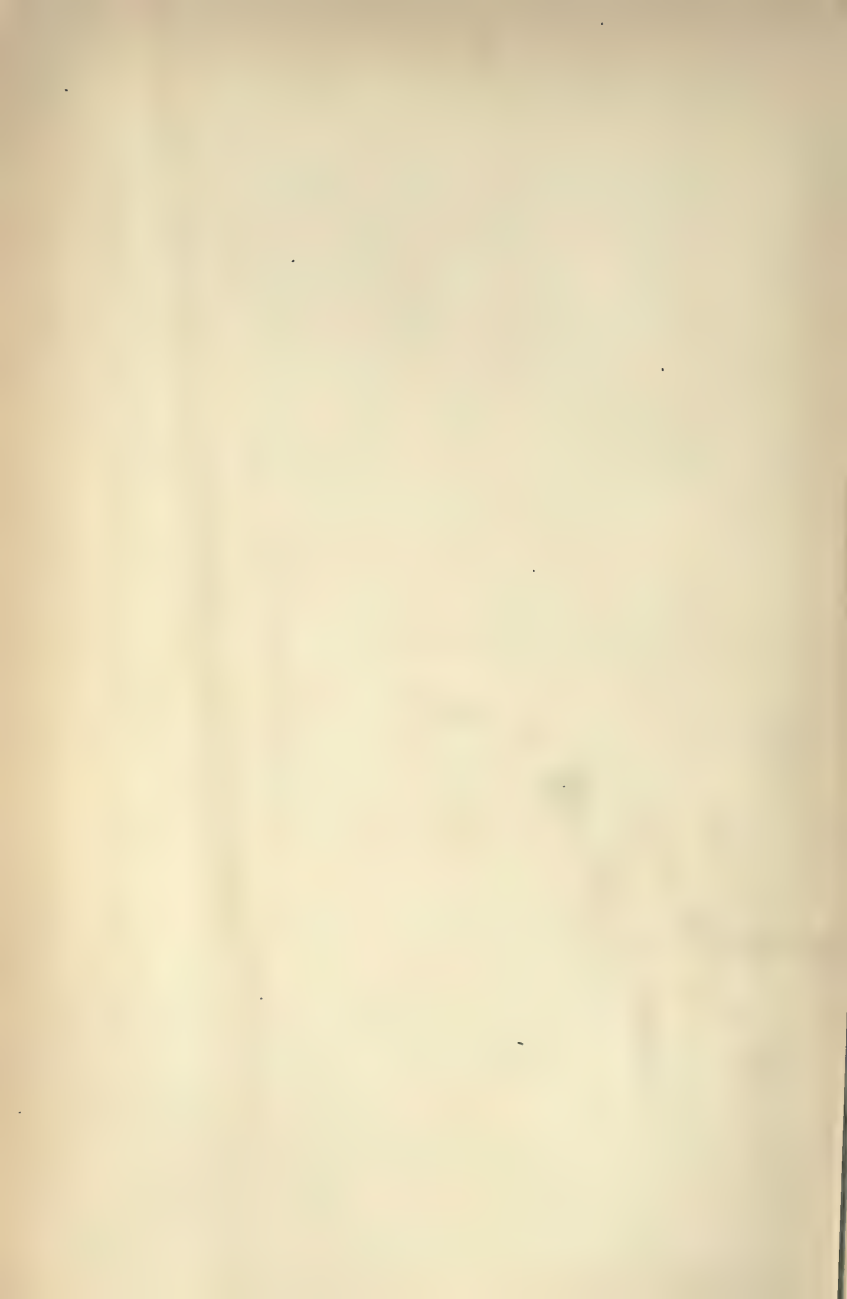
**7. REACTION OF SOLUBLE CARBONATES: • PROPERTIES OF CO<sub>3</sub> IONS (CARBONANION).** Add sodium carbonate solution to dilute solutions of barium chloride (?), lead nitrate (?), chromic chloride (?), cupric sulphate (?). Add the carbonate cautiously at first, note the gas given off in some cases and account for its formation. The gas is often slow to appear (why?). Filter the contents of each test-tube and wash each precipitate thoroughly (*cf.* p. 85, 4, *f*, note) with water. What are those precipitates? Test your conclusions by a suitable experiment in *each* case.

Save part of the lead compound and, when it is dry, heat it in a dry test-tube and ascertain what gas is given off (?). What carbonates show this behavior [R]?

**8. PURIFICATION OF SODIUM CHLORIDE.** Wash some crude salt with water and then prepare about 150 c.c. of cold sat-







urated solution by grinding the salt for some time in a mortar with the water. Place it in a beaker and pass hydrogen chloride into the solution. Prepare this gas by placing a handful of common salt in a generating flask, covering it with concentrated hydrochloric acid and allowing concentrated sulphuric acid to fall into it from a dropping-funnel. Deliver the gas into the solution through a thistle tube with the mouth downwards (why use this tube)? When considerable precipitation has occurred, filter by putting a clean silver coin in a funnel, pouring the liquid and crystals upon it and pressing with a spatula.

Explain why the salt is precipitated. If sodium sulphate or magnesium chloride or some other salt has been mixed with the salt, would they have been thrown down [R]? In other words, why does this process give a means of purification?

Explain the method of generating hydrogen chloride used above.

This question may be answered after the experiments in 9.

#### 9. IONIC EQUILIBRIUM.

*a.* Dilute a few drops of methyl orange solution with distilled water. Add to it first a few drops of an acid (?) and then a few drops of a base (?).

Take three portions of distilled water and add to each a little methyl orange solution. To the first two add a little acetic acid (?), to the third a drop or two of hydrochloric acid (?). What kind of ions is absent when the indicator is yellow? Add some solid sodium chloride to the first and stir (?). To the second add solid sodium acetate and stir (?). Explain the difference in behavior. To the third add solid sodium chloride and stir (?). Explain absence of effect.

*b.* Take three portions of a saturated solution of potassium chlorate in as many test-tubes. (This and the following solutions must be shaken to insure saturation if there is a deposit in the bottles.) To the first add saturated sodium chloride solution (?), to the second saturated potassium chloride solution (?), to the third saturated sodium chlorate solution (?). Allow them to stand for a minute or two before drawing any conclusion. Explain. The experiments will fail if the solutions are not saturated.

**10. REACTIONS OF SODIUM SALTS.** Try the flame test and examine with the spectroscope (?). Add tartaric acid (?) and picric acid (?) solutions to separate portions of diluted sodium chloride solution. Compare with results under potassium.

**11. AMMONIUM SALTS.** What is the effect of heating ammonium salts (p. 36, 4, *d*)? Heat some ammonium phosphate in a hard glass test-tube (?). Dissolve the residue in water and test with litmus paper. Heating and searching for an odor of ammonia is not a sure test for ammonium salts. In many cases the odor would not be perceived.

Try the flame test with an ammonium salt (?).

To two portions of ammonium chloride solution add excess of tartaric acid solution (?) and picric acid solution (?) respectively. What other ion gives the same results as ammonium with these reagents? To a third portion add a strong base and warm (?). Notice the odor. This is the best test for ammonium salts. How could you distinguish between solutions containing ammonium and potassium salts?

Make a list of the salts of potassium, sodium, and ammonium which are least soluble [*R* and *exps.*].







## CHAPTER XX.

### METALS OF THE ALKALINE EARTHS.

1. Heat 2-3 g. of powdered marble for fifteen minutes in an open porcelain crucible, with a blast-lamp (?). Add a little water to the product when it has cooled (?). (Has water any effect on marble?) Test the reaction of the liquid with litmus. What was formed by heating marble? The action is reversible. (How is this suggested by the behavior of mortar?) On what will the re-formation of marble depend at any given temperature [R]?

2. LIME WATER. Slake a small piece of calcium oxide and shake the product with half a liter of distilled water, let the solution settle, and use the clear liquid.

*a.* Blow air from the lungs by means of a tube through a part of the lime water (?). How could you determine the proportion of carbon dioxide in a sample of air?

*b.* Pass carbon dioxide from a Kipp's apparatus persistently through the remainder of the lime water (?). Boil a part of the resulting clear solution (?). Explain [R].

3. REACTIONS OF CALCIUM SALTS. Use calcium chloride solution and dilute it for *b*, *c*, and *d*. [In this and all following paragraphs headed "reactions," where diluted solutions are spoken of, the strong solutions on the side-shelf must be diluted with three to four times their volume of water to secure good results.]

*a.* Try the flame test and examine with the spectroscope (see that the platinum wire is clean). Make a sketch of the spectrum showing the positions of the lines with reference to the sodium and potassium lines.

*b.* To a solution containing calcium ions add ammonium carbonate solution, and warm if necessary.

*c.* To another portion of the solution add oxalic acid solution (?). [Formation of all precipitates, if long delayed, may be hastened by vigorous stirring with a glass rod.]

*d.* To another portion add excess of dilute sulphuric

acid (?). Filter, and neutralize the filtrate roughly with ammonium hydroxide. To this solution add ammonium oxalate solution (?), and explain (p. 28, 7, *d* and p. 52, 7, *a*). Is the sulphate or oxalate of calcium more soluble?

**4. REACTION OF STRONTIUM SALTS.** Use strontium chloride solution and dilute it for *b* and *c*.

*a, b.* Same as in 3.

*c.* To a portion of the strontium chloride solution add a solution of calcium sulphate (?) made by shaking a little of the powdered salt with water and decanting, and explain. The precipitation may be very slow.

**5. REACTIONS OF BARIUM SALTS.** Use barium chloride solution and dilute it for *b*, and *c*.

*a, b.* Same as in 3 and 4.

*c.* To a portion of the solution of barium chloride add a solution of strontium sulphate (?) made by shaking the salt with water and decanting, and explain.

Arrange the sulphates of these three metals in order of solubility. How could you tell a solution containing the ions of a member of this family from one containing those of the previous family?

Give two methods of distinguishing between the members of the present group.

**6. INSOLUBLE SULPHATES.** To a little lead nitrate solution add dilute sulphuric acid (?). Which sulphates have you found to be insoluble? These complete the list, as far as common metals are concerned.

**7.** Take three dry test-tubes, apply to the instructor for three "unknown" substances, and ascertain what each is, by the use of any experiments you can devise.

**SUGGESTIONS.** Study:

(1) Physical appearance (?).

(2) Odor (?).

(3) Solubility in water and reaction of the solution toward litmus (?).

(4) Effect of heating in a dry test-tube (?). Save the residue, as, after next experiment, examination of this may be necessary.

(5) Effect of heating with concentrated sulphuric acid (?).

Before trying the last two tests, make a list of all the gases which may be expected, the means of identifying each,





and the corresponding inferences with regard to the unknown substance. Test gases given off according to circumstances. The result of these experiments will suggest further work. The metals may be identified by the reactions given in Chaps. XIX and XX.

Make sure that your experiments and reasoning, which should be carefully written out, prove that the substance is the one you finally decide that it is, and exclude the possibility of its being any other. Report the result to the instructor.

## CHAPTER XXI.

### COPPER AND SILVER.

1. CUPROUS CHLORIDE [Hood]. Dissolve about 5 g. of copper clippings in warm *aqua regia*, using the minimum of nitric acid that will effect the solution of the metal. Why is the nitric acid required, and what other substances might serve the same purpose? Evaporate to dryness on the steam bath [Hood] and re-dissolve in 25 c.c. of water. Transfer to a flask, add an equal volume of concentrated hydrochloric acid and about 10 g. of copper clippings and boil [Hood] gently until the green tint is no longer perceptible in the dirty yellowish-brown color of the product (?). If a few drops added to a test-tube full of water confer a blue tinge on the solution the action is still incomplete.

To a small part of this solution, when cold, add sodium hydroxide solution (?). Why is so much of this required? Divide the mixture into two parts. Notice whether the precipitate undergoes any change on shaking with air (?). For comparison, add sodium hydroxide solution to cupric sulphate solution (?). Explain. Heat the other half (?).

Pour the rest of the cuprous chloride solution into a large amount of water in a beaker (?). Expose some of the substance while covered with water to the sunlight (?). What properties of cuprous chloride have you observed?

#### 2. DOUBLE SALTS.

a. Saturate water at 70° with 5 g. of finely powdered potassium sulphate (about 25 c.c. will be required). Calculate the weight of crystallized cupric sulphate which must be taken to get an equi-molecular proportion, and dissolve it in its own weight of hot water. Mix the two solutions, taking care not to allow any undissolved fragments of either salt to get into the mixture, and set the result aside to crystallize (?). Examine the form of the crystals and compare with those of blue vitriol (?). Keep them for use later.

b. To a little cupric sulphate solution add potassium cyanide solution till no further change occurs (?) [R]. Keep this solution.

After applying to these two preparations the tests in 4, c







and *d*, state the nature of the difference between the two bodies here examined.

**3. EQUIVALENT OF COPPER [Quant.].** Take a small rod of pure zinc, smooth ends with a file and weigh carefully. Place in a beaker an exactly known weight of crystallized cupric sulphate (about 2 g.), and dissolve in distilled water. Put the zinc in this solution and allow them to remain in contact until the latter is completely decolorized. Remove the zinc, free it carefully from the brown deposit (?), and dry and weigh it. What weight of zinc has gone into solution?

To avoid weighing the precipitate of copper, which it would be difficult to do exactly, calculate from the formula what quantity of copper was contained in the amount of blue vitriol taken (?). Calculate the equivalent weight of copper (that of oxygen being 8), using the value for the equivalent of the zinc found in Chap. V, 2, *b* or 3, *a* (p. 17) or, if zinc was not then employed, assume it to be 32.7. Look up the specific heat of copper (p. 20) and find its atomic weight from the equivalent observed.

What other atomic weights could be measured on this plan?

**4. REACTIONS OF CUPRIC SALTS.** Use diluted cupric sulphate solution. What is the color of cupric sulphate itself? To what is the color of the solution due?

*a.* Test the reaction of the solution with neutral litmus paper (?) and explain.

*b.* Add ammonium hydroxide till no further change occurs (?). This is used as a test for salts of copper.

*c.* Pass hydrogen sulphide gas through another portion. Make a solution of part of the crystals in 2, *a*, and apply this test to it (?) and to a part of the solution from 2, *b* (?).

*d.* Same as *c*, using potassium ferrocyanide solution in place of hydrogen sulphide. Answer the question at the end of 2.

*e.* Add potassium iodide solution to a fresh portion (?). Filter, wash the precipitate (?), and add a drop of the filtrate to starch emulsion (?).

*f.* Make a borax bead and heat it with a minute particle of cupric oxide in the oxidizing (?) and in the reducing (?) flame. The latter requires patience.

*g.* Try the match test (p. 74, 14) with any copper compound.

*h.* Boil a little of a dilute sugar solution with a few drops

of sulphuric acid for a minute or two. Add cupric sulphate solution and excess of sodium hydroxide solution and warm (*cf.* 1) [R].

#### 5. REACTIONS OF SILVER SALTS.

*a.* Take some silver nitrate solution and add to it dilute hydrochloric acid till no further precipitation occurs. Filter and wash with water.

What effect does the skin have on silver nitrate?

*b.* Treat part of the precipitate with ammonium hydroxide (?). Then add dilute nitric acid to the solution (?).

*c.* Place the rest of the precipitate in a porcelain crucible, put on it a piece of granulated zinc, and fill up with dilute sulphuric acid. Stir from time to time (?). After an hour or two pour off the acid, take out any unchanged zinc, wash the precipitate with water by decantation, add ammonium hydroxide, and filter. Find out whether there is any silver chloride in the filtrate (? *cf.* *b.*). When the filter paper is dry, place the dark powder in a hollow on a stick of charcoal and melt it with the flame of the blast-lamp directed downward upon it (?).

*d.* To a little silver nitrate solution add some potassium dichromate solution (?). Test the solutions before and after mixing, with neutral litmus paper (?). [If the color of the dichromate obscures that of the litmus, wash the test-paper with distilled water.]

**6. THE PROPERTIES OF A METALLIC ELEMENT.** The properties of a metal, in the chemical sense of the word, are: (1) The element may be the sole constituent of a cation (positive ion). (2) Its hydroxyl compound is a base. (3) Its salts should not be hydrolyzed by water.

Do copper and silver completely fulfil these conditions? If not, in what respects do they fail to do so?







## CHAPTER XXII.

### MAGNESIUM, ZINC, CADMIUM, MERCURY.

#### 1. PROPERTIES OF MAGNESIUM COMPOUNDS.

*a.* Try whether magnesium chloride dissolves completely in water(?). Test the solution with litmus(?).

Heat some of the crystals strongly in a dry test-tube(?). Test the reaction towards litmus paper of the water which condenses in the tube(?), and then remove the liquid from the sides of the tube with a piece of filter paper. Does the residue dissolve in water? Explain.

*b.* To some diluted magnesium sulphate solution add ammonium hydroxide(?). Explain the result in terms of the theory of ionization. Now mix with some ammonium hydroxide several times its volume of ammonium chloride solution (what effect will this have on the ionization of ammonium hydroxide?) and then add the mixture to a fresh portion of the magnesium sulphate solution(?). Explain. To this combination of three solutions add sodium phosphate solution(?) [R]. Write the equation and explain the purpose for which each ingredient was used.

*c.* To a fresh portion of the diluted magnesium sulphate solution add ammonium carbonate solution and warm(?). What other metal ions were precipitated by the same reagent? Repeat, adding excess of ammonium chloride solution to the magnesium sulphate solution before using the carbonate(?). Try whether, with this modification, the salts of those other metals still behave like those of magnesium. If you had a salt of magnesium mixed with a salt of one of those other metals, how would you proceed so as to precipitate a compound of the alkaline earth metal first and one of magnesium afterwards? Explain the effect of the ammonium chloride as in *b*.

Add two drops of hydrochloric acid (why?) to about 250 c.c. of the city water, evaporate to small bulk, and test it for calcium and magnesium.

*d.* Pass hydrogen sulphide through some magnesium sulphate solution(?).

2. SALTS OF ZINC. Use diluted zinc sulphate solution for *b*, *c*, and *d*.

*a.* Relative activity of acids. In three clean test-tubes place (1) zinc chloride, (2) zinc sulphate, and (3) zinc acetate solutions. Test each with litmus paper(?). Pass hydrogen sulphide to saturation (test?) through each solution(?). Filter the mixtures separately, test the reaction of each filtrate with litmus paper(?), and add ammonium hydroxide to each(?). In considering the nature of the product produced by the last reagent, remember that the water is saturated with hydrogen sulphide, and that therefore ammonium sulphide is formed in it.

How do the three salts differ in behavior? How do you account for this difference? In answering this question it will be found helpful to scrape a little of the precipitate into each of two test-tubes and to treat one with diluted hydrochloric acid (?) and the other with diluted sulphuric acid(?). What evidence is there that the actions are reversible? Which shows this most markedly and which least so? Can you infer from this the relative activities of the three acids? Preserve some of the precipitate for use in *d*.

*b.* Ionic equilibrium. Take a larger amount of zinc sulphate solution and add sulphuric acid to it cautiously until a sample ceases to give any precipitate with hydrogen sulphide. Explain. Now add much powdered solid sodium sulphate, stir until it has dissolved, and test a part with hydrogen sulphide again. Explain. Write this equation so as to show that the action is reversible and that an equilibrium exists.

*c.* To zinc sulphate solution add caustic soda solution until no further change occurs (?). Explain [R].

*d.* Take a piece of filter paper from *a* with a very little zinc sulphide on it, roll it up, and twist the platinum wire tightly round it. Roast the whole in the Bunsen flame. Moisten the ash with cobalt chloride solution and heat again (?).

**3. CADMIUM SALTS.** Take two samples of a diluted solution of any cadmium salt. Test the reaction with litmus. To the first add hydrogen sulphide gas (?). To the second add sodium hydroxide solution in excess (?). Acidify the result of the first with hydrochloric acid (?). How do the salts of this metal differ in behavior from those of zinc?

By what reactions could you distinguish between salts of magnesium, zinc, and cadmium?

**4.** Apply to the instructor for three unknown substances and identify them.





**5. MERCUROUS NITRATE.** Place about 10 g. of mercury with 15 c.c. of diluted (1:1) nitric acid in a small beaker and let the action go on for an hour or two, or until fresh crystals cease to be formed. If crystallization is long in starting, stirring, or infection with a crystal of mercurous nitrate will bring it about. Pour away the liquid and dissolve the crystals in water to which a few drops of nitric acid have been added (why?). This solution, if ready, may be used in **6**.

How could you make mercuric nitrate solution?

**6. REACTIONS OF SALTS OF MERCURY.** Use diluted portions of mercurous nitrate solution and of a solution of any mercuric salt and add to each the following reagents. Compare results in each case.

- a. Litmus (?).
- b. Dilute hydrochloric acid (?). Treat the precipitate, if there is any, with ammonium hydroxide (?) [R]
- c. Hydrogen sulphide to saturation (?), then acidify (?).
- d. Ammonium hydroxide (?) [R]
- e. Sodium hydroxide (?).
- f. Potassium iodide till there is no further change (?).
- g. Stannous chloride till there is no further change (?).
- h. Clean copper clippings (?). Ascertain experimentally whether any copper goes into solution (?).
- i. Heat any salt of mercury strongly in a narrow tube closed at one end (?).

How could you distinguish a solution of a mercurous and of a mercuric salt, respectively, from salts of silver, bismuth, magnesium, zinc, and cadmium?

**7.** Do these four metals exhibit the properties of metallic elements (p. 94, 6)? If not, in what respects do they fail to do so?

## CHAPTER XXIII.

### ALUMINIUM, TIN, LEAD.

#### 1. ALUMINIUM.

*a.* Recall the effect of hydrochloric acid on aluminium (p. 22, 1, *a*) (?). Try diluted nitric acid (?).

*b.* Heat a piece of aluminium wire with sodium hydroxide solution for some minutes (?). To ascertain whether anything has gone into solution, neutralize carefully with dilute hydrochloric acid (?). Neutralize a sample of the caustic soda solution employed (?). If there is a precipitate in either case test it by 3, *e*.

**2. ALUM.** Prepare warm saturated solutions of anhydrous aluminium sulphate and ammonium sulphate in approximately equi-molecular proportions, mix them, and set aside (?). Obtain some large crystals by hanging a thread in the solution. Notice the form of the crystals.

Ascertain by experiments selected from 3 whether a solution of this salt behaves, in respect to the aluminium which it contains, like a mixture of the constituent salts or like a different salt. Is alum a double salt or a salt of a complex acid?

**3. REACTIONS OF ALUMINIUM COMPOUNDS.** Use a diluted aluminium sulphate solution.

*a.* Test the solution with litmus (?).

*b.* Add sodium carbonate solution (?). Isolate the precipitate and find out whether it is a carbonate or not.

*c.* To another portion add colorless ammonium sulphide (?). Find out experimentally whether the precipitate is a sulphide or not (?). Preserve part of the precipitate for use in *e*.

*d.* To another portion add sodium hydroxide solution gradually (?). Filter, suspend part of the precipitate in water, and treat with more sodium hydroxide solution (?). What other hydroxide behaves like this? Treat another part of the precipitate similarly with hydrochloric acid (?). What peculiarities does this hydroxide show?

*e.* Wrap up part of the filter paper from *c*, twist the plat-







inum wire tightly round it, char in the Bunsen flame, moisten with cobalt chloride solution, and heat again (?).

f. To some cochineal solution add any solution containing aluminium sulphate and then ammonium hydroxide (?). Filter. Repeat the treatment with aluminium sulphate and ammonium hydroxide, if necessary (?).

#### 4. HALIDES OF TIN.

a. Stannous chloride. Dissolve tin in warm concentrated hydrochloric acid. Let the action go on until the acid is nearly exhausted. Use the solution in 4, b, and 5. Proceed with 6, 8, and 9 until it is ready.

b. Stannic halide [Hood]. To part of the solution from a add bromine until the color ceases to be destroyed and drive off the excess of bromine by warming (?). Use this liquid in 5.

5. REACTIONS OF STANNOUS AND STANNIC SALTS. Use a portion of each of the above solutions, after diluting as usual, with each reagent.

a. Saturate (test?) each solution with hydrogen sulphide (?). Neutralize with ammonium hydroxide and add yellow ammonium sulphide to each product (?). Filter and acidify each resulting solution with hydrochloric acid (?).

b. Add mercuric chloride solution to fresh portions of each (?). Boil a portion of the stannic halide with a piece of tin for a minute or two and test with mercuric chloride solution again (?).

c. Add sodium hydroxide solution to each until no further change occurs (?). What peculiarity do these hydroxides exhibit?

6. SOLUTION TENSION AND ION CONCENTRATION. Suspend a rod of tin about 60 mm. long by a thread from one end, and hang it near the bottom of a wide test-tube or narrow cylinder. Pour in through the dropping funnel, which must reach the bottom of the cylinder, first highly diluted dilute hydrochloric acid (1:3) and then diluted (1:1) stannous chloride solution. Perform the operation with care, in such a way that the solutions do not mix and that the surface at which they meet is near the middle of the rod of tin. If the second solution is permitted to carry air bubbles with it, mixing will inevitably occur. Place the arrangement where it will not be disturbed, and examine it from time to time (?). Explain [R].

7. Apply to the instructor for two unknown substances and identify them.

**8. LEAD.**

*a.* Dissolve 1 g. of lead acetate in 20 c.c. of water, place in it several pieces of granulated zinc and let them remain for an hour or two. Preserve the solution and, after **9**, devise a way of precipitating any remaining ionic lead and showing the presence of zinc in the solution, and see whether it works.

*b.* Wash some of the lead from *a* with distilled water and see whether it is possible to get washings which show no reaction with hydrogen sulphide (?). Account for what you observe.

**9. REACTIONS OF LEAD SALTS.** Use diluted lead nitrate solution.

*a.* Test the solution with litmus (?).

*b.* Hydrogen sulphide (?).

*c.* Hydrochloric acid (?). Filter, dilute the filtrate, and pass hydrogen sulphide through it (?). Explain. What other chlorides are more or less insoluble in water?

*d.* Potassium iodide solution (?). Boil the result, filter, and examine the filtrate (?). What property of plumbic iodide is indicated?

*e.* Add sodium hydroxide gradually (?) and then in excess (?). Compare the behavior of the hydroxide with that of hydroxides of zinc, aluminium, and tin.

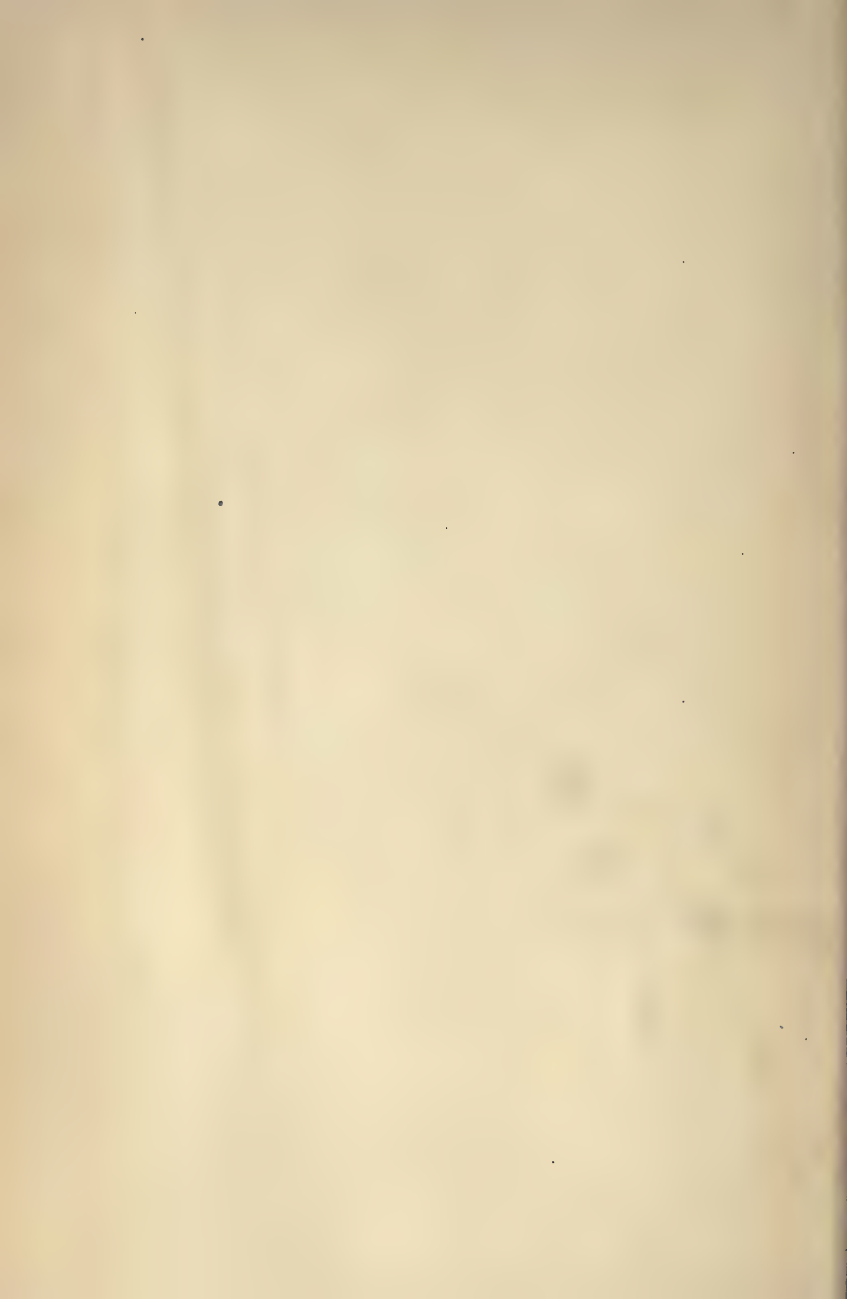
*f.* Potassium dichromate solution (?). Test the solutions with litmus paper before and after mixing (?). The result will throw light on the nature of the action [R].

Does lead form any compounds in which it is quadrivalent [R]?

Explain the action of minium on hydrochloric acid (p. 30, 1, *a*).

**10.** Do these three metals exhibit the properties of metallic elements (p. 94 6)? If not, in what respects do they fail to do so?







## CHAPTER XXIV.

### CHROMIUM, MANGANESE.

**1. CHROMIC OXIDE.** Mix some potassium dichromate (15 g.) thoroughly with one-fifth its weight of powdered sulphur and heat with the blast-lamp in a porcelain crucible for fifteen minutes. Grind up the resulting mass in a mortar with water, filter, wash the green residue (?), and dry it on a radiator for use in **2**.

Make a borax bead, dissolve a particle of chromic oxide in it, and note the effects of the oxidizing and reducing flames on it (?). All chromium compounds give the same result. If chromic sulphate had been used, what would have been the nature of the chemical action?

**2. CHROMIC CHLORIDE.** Mix the chromic oxide prepared in **1** with one-third its weight of powdered wood charcoal, make into a stiff paste with some starch, and mold the mixture into little pellets of the size of peas. Cover these completely with a layer of charcoal powder (why?) in a closed crucible, dry them by heating with the Bunsen flame and let them cool before exposing them to the air (why?). Place them in a piece of hard glass tubing. Then connect with a chlorine apparatus and, when the chlorine gas has reached the pellets and completely displaced the air (why?), heat strongly with a blast-lamp. Conduct any superfluous chlorine into a test-tube filled with sodium hydroxide. Describe the substance which is formed and try its solubility in water and acids.

**3. CHROME-ALUM.** Dissolve 10 g. potassium dichromate in water, add the amount (calculated) of sulphuric acid necessary to form potassium sulphate and chromium sulphate, warm and add alcohol (7-10 c.c.), a little at a time, until the yellow color has entirely given place to a pure, bright green. The action takes some time to reach completion. Notice the odor (?). Set the solution aside to evaporate spontaneously. Examine the form and color of the crystals when they appear. What is the color of their solution in water?

**4. CHROMATES.** Melt 5 g. potassium carbonate with equal amounts of potassium hydroxide and potassium nitrate at a

low temperature in an iron crucible and stir in (use the reverse end of a file) 5 g. of powdered chromite. Heat strongly [Blast-lamp] for several minutes (?). When the mass has cooled dissolve it in a little boiling water. Add dilute nitric acid until the solution is acid (?). Note the change in color (?).

5. CHROMIC ACID. Make a cold saturated solution of sodium dichromate, add it to two volumes of concentrated sulphuric acid and cool (?). Filter through a small plug of asbestos and dry the precipitate by smearing it on a piece of broken bisque plate [Storeroom].

6. Take some potassium dichromate solution and run into it potassium hydroxide solution from a burette till the change in color is complete. A test-tube trial will show the tint to be reached. Concentrate the solution and allow it to crystallize (?). What kind of salt (neutral, acid, basic, double, or complex) is potassium dichromate essentially?

7. REACTIONS OF CHROMIC SALTS. Use diluted, freshly prepared chrome-alum solution. What are the ions in the solution?

a. Boil one portion of the solution for some time [R]? What was the original color of the solution?

b. To another portion add sodium hydroxide solution, at first a little (?), then excess (?). Boil.

c. Add colorless ammonium sulphide (?). Is the precipitate a sulphide?

d. Add excess of sodium hydroxide solution and then a large volume of bromine water, and heat (?). Try another portion using lead dioxide instead of bromine (?). Infer the nature of the action from the change in color.

8. REACTION OF CHROMATES. Use diluted potassium chromate solution. What are the ions in the solution?

a. Acidify the solution with dilute nitric acid (?).

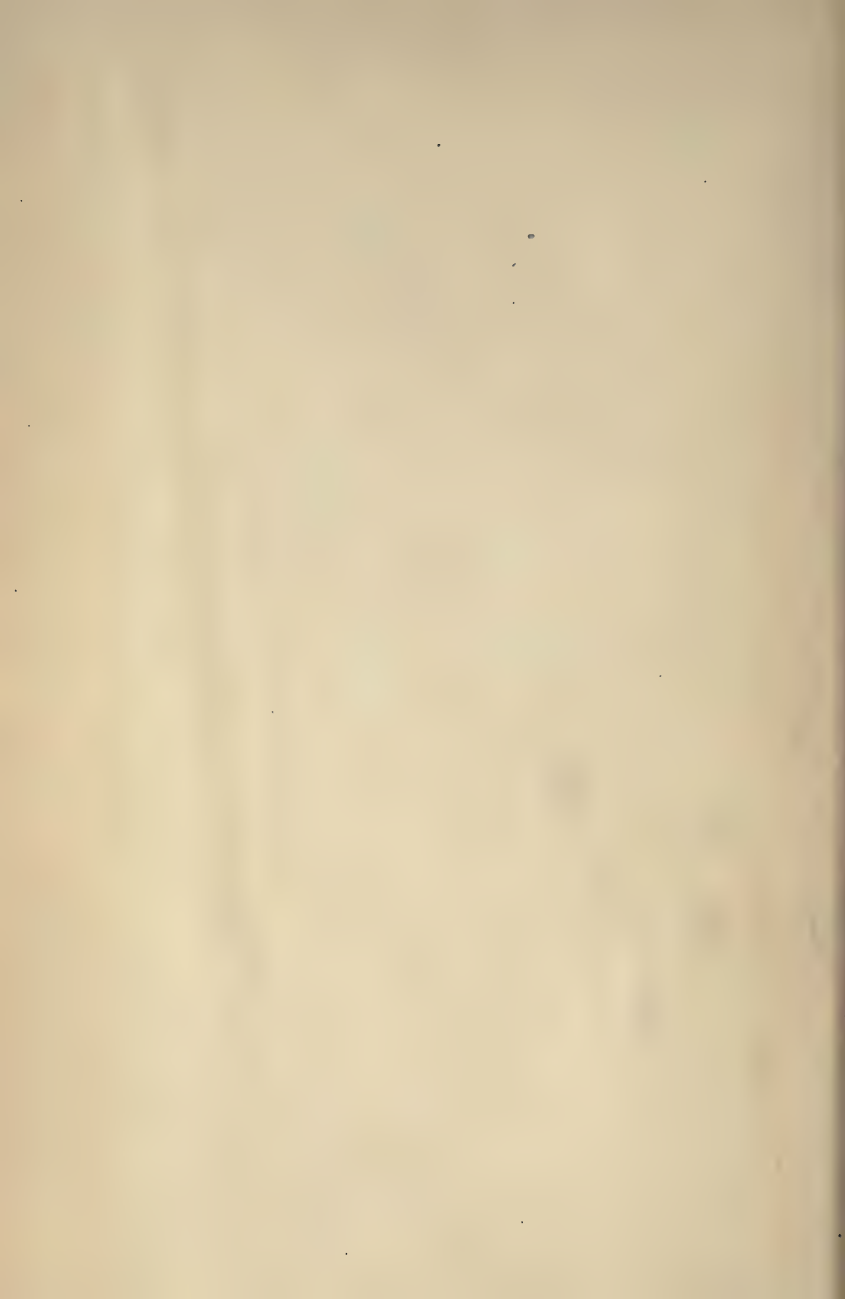
b. Recall the actions of hydrogen sulphide, of sulphur dioxide, and of hydrogen peroxide, on such an acid solution (?).

c. Add colorless ammonium sulphide, heat and maintain at the boiling point, noting two distinct changes (?), then acidify (?).

d. Add lead nitrate and barium chloride to separate portions.

9. MANGANOUS AND MANGANIC SALTS. Recall the preparation of a manganous salt on p. 30, 1, b. Name a manganic salt. What are the colors of their solutions and therefore of the





characteristic ions [R]? The chemical actions in 10 can be followed by noting the changes in color.

#### 10. MANGANATES AND PERMANGANATES.

*a.* Fuse a mixture of 5 g. of potassium hydroxide, 2.5 g. potassium chlorate, and 5 g. finely powdered manganese dioxide at a red heat, stirring with the reverse end of a file, until effervescence ceases(?). Add the last ingredient gradually. Treat the mass with a small amount of cold water, decant the clear liquid away from the precipitate, and use it in *b*, *c*, and *d*.

*b.* Dilute a part of the clear green solution, with a very large amount of water in a beaker(?). If no change should occur, pass carbon dioxide into the diluted solution(?).

*c.* To a portion of the green solution add a few drops of alcohol and warm(?).

*d.* To the rest of the green solution add a boiling solution of oxalic acid(?).

*e.* Repeat *c* and *d* with potassium permanganate solution, acidified by adding two or three times its volume of dilute sulphuric acid(?).

*f.* Add similarly acidified permanganate to a little diluted ferrous ammonium sulphate solution till the pink color is permanent(?). To the product add ammonium hydroxide(?). What change has taken place in the iron (p. 69, 4, *c*)?

*g.* Recall the action of the same reagent on hydrogen sulphide (p. 57, 3, *d*) and on sulphurous acid (p. 62, 9, *d*).

#### 11. REACTIONS OF MANGANOUS SALTS. Use any manganous salt.

*a.* Borax bead in oxidizing(?) and reducing(?) flames.

*b.* Bead of a mixture of sodium carbonate and sodium nitrate on a platinum wire with any manganese compound(?).

*c.* To a diluted solution of a manganous salt, add ammonium sulphide(?). Is the product a sulphide?

*d.* To another portion add sodium hydroxide(?). Divide into two parts. Shake one with air(?). To the other add bromine water and warm(?).

#### 12. Apply to instructor for two unknown substances and identify them.

## CHAPTER XXV.

### IRON, COBALT, NICKEL.

1. IRON. Recall the action of iron on dilute acids (p. 22, 1, *a, c, d*), also its preparation from the oxide (p. 24, 5, *a*).

#### 2. FERRIC SULPHATE.

*a.* Weigh 3.5 g. of concentrated sulphuric acid into an evaporating dish, add 20 g. of crystallized ferrous sulphate, and dissolve in 25–30 c.c. of water. Heat on a square of wire gauze [Hood] and add concentrated nitric acid drop by drop till the color of the solution, dark at first (?), changes to a light brown (? p. 69, 4, *c*). Evaporate to a syrup on the steam bath, dissolve the residue in the minimum amount of boiling water, add 3.5 g. of ammonium sulphate, likewise dissolved in the minimum amount of boiling water, and set aside to crystallize. Describe the crystals (?). Collect them and wash them free from the mother liquor, dry with filter paper, and preserve for use in 4.

Chlorine, bromine, and other oxidizing agents have the same effect as the nitric acid here used. With what other have we previously produced the same change?

*b.* Dissolve some ferric sulphate in water. Note the color (?) and reaction of the solution (?). Add some pure sulphuric acid (?). Account for what you observe and explain the behavior of the salt in terms of the ionic theory.

3. SALTS OF COMPLEX ACIDS. Ferrocyanide and ferricyanide of potassium. Take some potassium ferrocyanide solution in a test-tube and add bromine water until a drop of the solution gives no blue precipitate with a dilute ferric chloride solution. What does the solution contain [R]?

#### 4. REACTIONS OF FERROUS AND FERRIC SALTS.

*a.* Borax bead with any compound of iron (the oxide or sulphate is best) in oxidizing and reducing flames (?).

Examine the action of the following substances on a diluted solution of ferrous ammonium sulphate (freshly prepared) and on a diluted solution of iron alum (made in 2) or ferric chloride.

*b.* Test the effect of each solution on litmus paper (?). Which has the stronger effect? What do we infer (p. 94, 6)







from this? If the difference is not plain, try paper dipped in "Congo red" [Storeroom].

*c.* To a portion of each solution add ammonium hydroxide(?). Note the effect on each precipitate of shaking with air(?).

*d.* To another pair of portions add ammonium sulphide solution(?) [R]. Is the precipitate soluble in hydrochloric acid? How could you prove experimentally that free sulphur is formed in the case of ferric salts?

*e.* Potassium ferrocyanide solution (?).

*f.* Potassium ferricyanide solution (?).

*g.* Potassium sulphocyanide solution (?).

*h.* The reduction (p. 58, 3, *e*), of ferric salts in solution is as easy as the converse operation (2, *a*). Boil a little diluted ferric chloride solution with a pinch of powdered iron and test a drop from time to time (?). What reagent will you use in testing?

*i.* Ascertain experimentally whether solutions of ferro- and ferricyanides contain a detectable proportion of iron ions.

*k.* Show that the crude hydrochloric acid contains ferric chloride (?). Show that the same substance contains sulphuric acid as another impurity. (The yellow color is partly due to the presence of an organic substance.)

**5. REACTIONS OF SALTS OF COBALT.** Use diluted cobalt chloride solution.

*a.* Borax bead in oxidizing and reducing flames (?).

*b.* Add sodium hydroxide solution—first a little (?), then excess, and warm (?).

*c.* Ammonium sulphide solution (?).

**6. REACTIONS OF SALTS OF NICKEL.** Use diluted nickel sulphate solution.

*a, b, c.* Same as in 5.

**7.** Obtain two unknown substances from the instructor and identify them.

## APPENDIX

### TENSION OF AQUEOUS VAPOR IN MILLIMETERS.

TEMP.	PRESS.	TEMP.	PRESS.	TEMP.	PRESS.
0°	4.6	16°	13.5	26°	25.1
5	6.5	17	14.4	27	26.5
8	8.0	18	15.4	28	28.1
9	8.6	19	16.3	29	29.8
10	9.2	20	17.4	30	31.5
11	9.8	21	18.5	31	33.4
12	10.5	22	19.7	32	35.4
13	11.2	23	20.9	33	37.4
14	11.9	24	22.2	34	39.6
15	12.7	25	23.6	100	760.0

### DEGREE OF IONIZATION OF ACIDS, BASES AND SALTS.

Except where otherwise specified, the figures give the percentage ionized in a normal solution at 18°, calculated from the electrical conductivities.

ACIDS	PER CENT.	SALTS	PER CENT.	BASES	PER CENT.
HNO <sub>3</sub> . . . .	82.0	KCl . . . .	75.0	LiOH . . . .	63
HNO <sub>3</sub> (conc., 62%)	9.6	NH <sub>4</sub> Cl . . . .	74.0	NaOH . . . .	73
HCl . . . .	78.4	NaCl . . . .	67.6	KOH . . . .	77
HCl (conc., 35%)	13.6	HgCl <sub>2</sub> . . . .	< 1.0	Ba(OH) <sub>2</sub> . . . .	69
HMnO <sub>4</sub> (N/2, 25°)	93.3	KNO <sub>3</sub> . . . .	64.0	Ca(OH) <sub>2</sub> (N/64,	
HI (N/2, 25°) .	90.1	K <sub>2</sub> SO <sub>4</sub> . . . .	53.0	25°) . . . .	90
HBr (N/2, 25°)	89.9	K <sub>2</sub> CO <sub>3</sub> . . . .	(49.0)	Sr(OH) <sub>2</sub> (N/64,	
H <sub>2</sub> F <sub>2</sub> . . . .	7.0	KClO <sub>3</sub> (N/2) . . . .	79.0	25°) . . . .	93
H <sub>2</sub> SO <sub>4</sub> . . . .	51.	Na.HCO <sub>3</sub> . . . .	(52.0)	Ba(OH) <sub>2</sub> (N, 64,	
H <sub>2</sub> SO <sub>4</sub> (conc., 95%)	0.7	Na <sub>2</sub> C <sub>4</sub> H <sub>4</sub> O <sub>6</sub> (N/32,		25°) . . . .	92
H.H <sub>2</sub> PO <sub>4</sub> (N/2,		25°) . . . .	(78.0)	AgOH(N/1783,	
25°) . . . .	17.0	ZnSO <sub>4</sub> . . . .	24.0	25°) . . . .	38.8
HC <sub>2</sub> H <sub>3</sub> O <sub>2</sub> . . . .	0.4	Hg(CN) <sub>2</sub> . . . .	< 1.0	NH <sub>4</sub> OH . . . .	0.4
HC <sub>2</sub> H <sub>3</sub> O <sub>2</sub> (N/10)	1.3	CaSO <sub>4</sub> (N/100)	63.0	H.OH < 1 per 10 mil.	
H.HCO <sub>3</sub> (N/10)	0.17				
H.HS(N/10) .	0.07				
H.H <sub>2</sub> BO <sub>3</sub> (N/10)	0.01				
HNC(N/10) .	0.01				

SELECTION FOR STUDENTS WITH ADMISSION  
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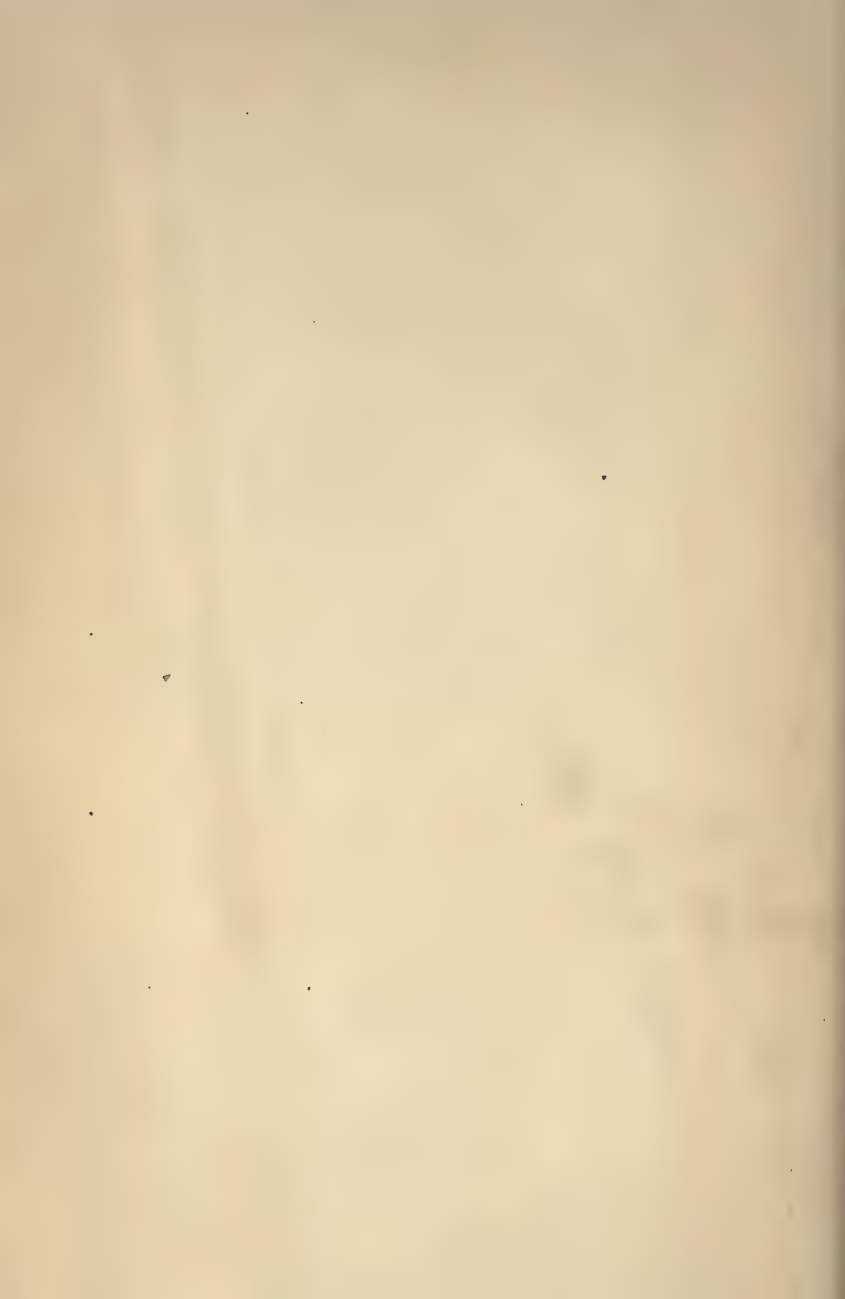
## CHAP.

- I. 1; 2; 3; 5; 6.  
III. 1, *b*; 2, *a* or *b*.  
IV. 4; 5; 6.  
V. 2, *a*; 3 *a*; 5 or 6 *a* or *b*; 7.  
VI. 1; 2, *c*, *d*; 5.  
VII. 2; 3, *d*; 6; 7.  
VIII. 1, *a*, 3, *a*, *b*, *c*.  
IX. 2; 4, *d*.  
X. 1; 2, *a*, *b*, *d*; 3, *a*,  
4; 5; 6; 7; 8.  
XI. 2; 3; 4; 5; 6; 7.  
XII. 1; 2, *d*, *e*; 3; 6; 7; 8;  
9; 10.  
XIII. 2, *c*; 3; 4; 5, *c*;  
9; 10; 11; 12.

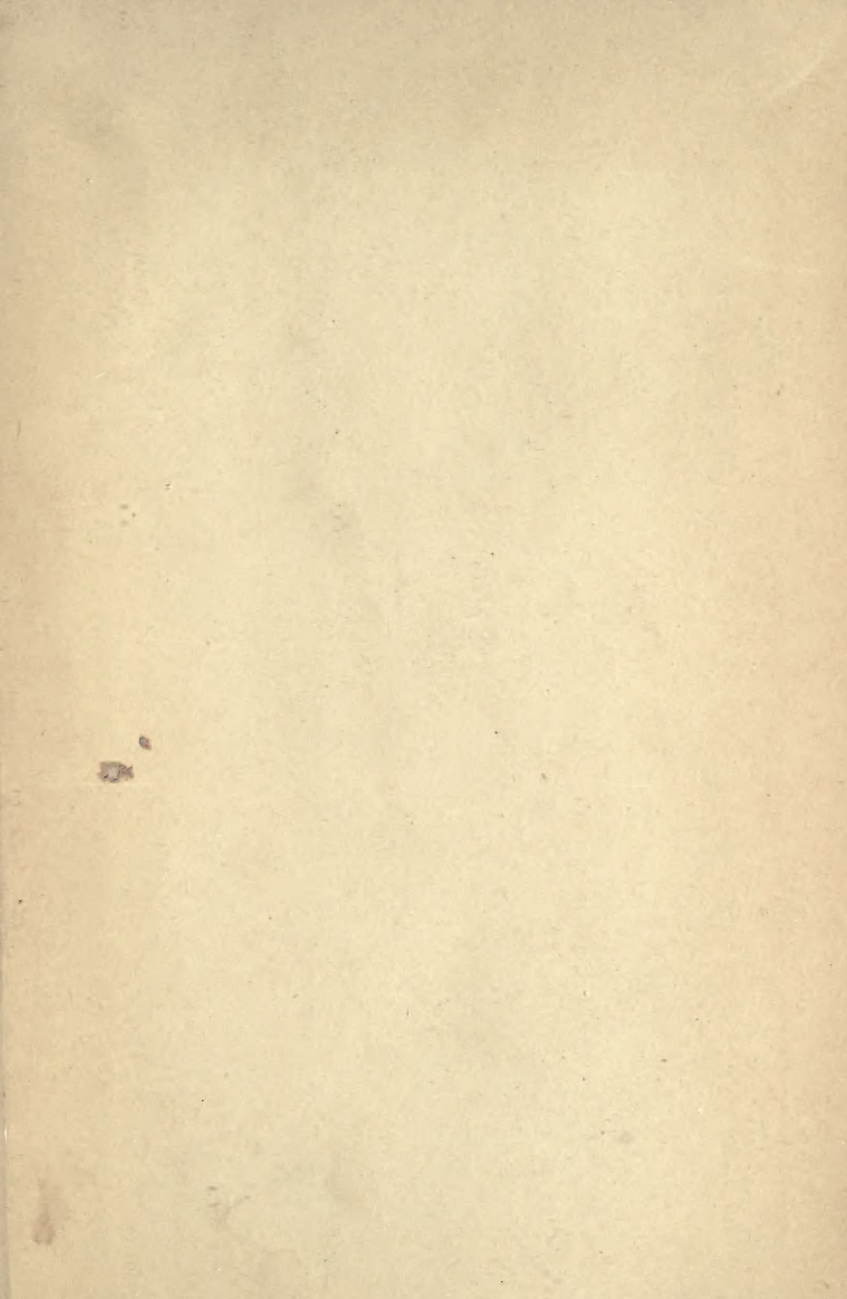
## CHAP.

- XIV. 1.  
XV. 1, *b*, *d*; 2; 3, *b*, *c*; 4; 5.  
XVI. 2; 3, *b*; 5; 6, *b*; 8; 9;  
12; 13.  
XVII. 8, *b*; 9, *b*.  
XVIII. 1; 3.  
XIX. 2; 3; 4; 6; 7; 8; 9.  
XX. 3; 4; 5; 7.  
XXI. 1; 2; 6.  
XXII. 1; 2; 3; 4; 5; 6; 7.  
XXIII. 1; 2; 4; 6; 8; 10.  
XXIV. 1; 2; 4; 5; 9; 10.  
XXV. 3; 4; 7.

This suggested selection of laboratory work for college students who have admission credit, on the basis of a year of chemistry in a secondary school, is subject to further adaptation to individuals. In all cases the student will be expected to master the topics omitted in the laboratory.









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